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HANDLING AND STORAGE OF NITROGEN TETROXIDE

TECHNICAL DOCUMENTARY REPORT NO. RTD-TDR-63-1033
MAY 63

PROJECT 8119 TASK 811906 (FORMERLY 6054 AND 60182)

(PREPARED UNDER CONTRACT NO. AF MIPR (33-616)60-20 BY THE ARMY CHEMICAL CENTER, EDGEWOOD, MARYLAND)

ROCKET PROPULSION LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
EDWARDS, CALIFORNIA

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ABSTRACT

A comprehensive source of basic information for use in the design, fabrication, and operation of nitrogen tetroxide handling equipment. The chemical and physical properties of this propellant are included so that the hazards will be recognized and understood. Principles underlying the prevention of fire, explosion, and toxic effects are presented, along with the information on the disposal and neutralization of vapors and liquids.

PUBLICATION REVIEW

The publication of this report does not constitute approval by the Air Force of the findings or conclusions contained herein. It is published only for the exchange and stimulation of ideas.

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HANDLING AND STORAGE OF NITROGEN TETROXIDE

1. INTRODUCTION.

The information contained in this manual is presented as a guide for the use of the engineer, operations supervisor or safety officer. The object is to provide technical personnel with a comprehensive source of basic information for use in the design, fabrication and operation of nitrogen tetroxide handling equipment.

The chemical and physical properties of this propellant are included so that the hazards will be recognized and understood. Principles underlying the prevention of fire, explosion, and toxic affects are presented, along with information on the disposal and neutralization of vapors and liquids.

Proprietary or copyrighted names of products are used whenever a description is complex or impractical. Such use is not to be construed as a preferential product endorsement by the Department of Defense or as an exclusion of other products found by test to be as good. Other products, however, should be tested prior to use.

In addition to the written text, a set of drawings for a typical bulk storage facility is furnished. These drawings provide the basic information necessary for the design, construction, installation, and operation of the facility.

Some of the data presented in this manual has been extracted from governmental and industrial literature sources. References to these sources are made throughout the text. A list of these documents is presented in paragraph 10, References.

2. GENERAL PROPERTIES. (Ref. 4)

2.1 Chemical Composition.

Nitrogen tetroxide is a corrosive liquefied gas and is extremely poisonous. This oxidizer is sometimes called dinitrogen tetroxide, nitrogen

peroxide, or liquid nitrogen dioxide. It is actually an equilibrium mixture of nitrogen tetroxide and nitrogen dioxide (N₂O₄ 2NO₂). Propeilant nitrogen tetroxide, specification MIL-P-26539(USAF) containing a minimum purity of 99.5% by weight and no more than 0.1% water equivalent, is sometimes referred to as dry nitrogen tetroxide.

2.2 General Appearance.

At room temperature, nitrogen tetroxide is a heavy brown liquid because of the NO₂ content. As the temperature is lowered, the color becomes lighter due to the equilibrium shift of NO₂ to N₂O₄. The fumes are yellowish to reddish brown, depending on the temperature, and have a characteristic pungent odor.

2.3 Chemical Nature.

Nitrogen tetroxide is a corrocive oxidizing agent. It is hypergolic with UDMH, hydrazine, analine, furfuryl alcohol, and some other fuels. It is not sensitive to mechanical shock, heat, or detonation. It is non-flammable with air; however, it can support combustion with combustible materials.

2.4 Physical Properties.

Boiling Point, ^o F	70.1
Freezing Point, OF	11.84
Density at 68°F, lb/gal	
Specific gravity of gas at 70°F, 1 atm	
Viscosity at 70°F, 1 atm, centipoise	0.413
Critical Temperature oF	316.8
Critical Pressure, psig	
Critical Density, lb/gal	

Vapor Pressure

Temp.,	or	psig
32		-9.6
70	,	0.1
90		10
100		16
120		34
140		59
160		97

2.5 Solubility.

Nitrogen tetroxide is soluble in water, forming nitric and nitrous acids. The nitrous acid undergoes decomposition forming additional nitric acid and evolving nitric oxide (NO). However, the degree of solubility depends upon how completely it can be exposed to water.

2.6 Stability.

Nitrogen tetroxide is very stable at room temperature. It begins to dissociate at 302°F into nitric oxide and free oxygen; however, upon cooling, it re-forms into nitrogen tetroxide. Dry (less than 0.1% water equivalent) nitrogen tetroxide may be stored and shipped in low pressure carbon-steel containers since the vapor pressure at 140°F is only 74 psia, and the corrosivity at this water content is negligible for an indefinite period.

3. HAZARDS.

3.1 Health Hazards.

- 3.1.1 General Nitrogen tetroxide in liquid form is injurious to body tissues. It volatilizes readily, giving off yellowish to reddish-brown fumes containing a mixture of nitrogen tetroxide (N₂O₄) and nitrogen dioxide (NO₂). Most discussions of the toxicity of these fumes identify the mixture as one compound or the other; likewise, calculations of atmospheric concentrations are normally made in terms of one compound or the other. This manual will follow the same practice; however, it should be kept in mind that the two oxides exist together in equilibrium.
- 3.1.2 Toxicity The liquid is corrosive, so severe burns of the skin and eyes can result from more than momentary contact, i.e., unless immediately removed. Inhalation of toxic vapors is normally the most serious hazard in the handling of nitrogen tetroxide. The threshold limit value, MAC, (maximum acceptable concentration) of the fumes is 5 ppm (9 mg/cu m) expressed as nitrogen dioxide, or 2.5 ppm (9 mg/cu m) expressed as nitrogen tetroxide. The main danger from acute poisoning is the development of pulmonary edema (a filling of the lung spaces with fluid) with resultant reduction of the ability of the lungs to transport oxygen. This condition normally

develops much later than the exposure to the fumes. A man may, without serious discomfort at the time, breathe an atmosphere containing a dangerous concentration of nitrogen tetroxide, and then hours later (sometimes as much as a day later) become severely ill. The color of the fumes is not a reliable index of degree of toxic hazard. The initial symptoms of poisoning--irritation of the eyes and throat, cough, tightness of the chest, and nausea--are slight and may not be noticed. Severe symptoms begin some hours later; their onset may be sudden, and precipitated by exertion. Coughing, a feeling of constriction in the chest, and difficult breathing occur. Cyanosis (a blue tinge to the mucous membranes of the mouth, eyelids, lips, and fingernail beds) may follow. Persons with such symptoms are in great danger. Milder cases may show signs of either bronchitis with cyanosis, or nausea, abdominal pain, and vomiting.

Repeated exposure to these fumes at low concentration levels may cause ulceration of the nose and mouth, wearing down and decay of teeth, and chronic irritation of the entire respiratory tract. Bronchitis, bronchiectasis, and secondary pulmonary emphysema may occur.

3.1.3 First Aid and Self-Aid - If liquid nitrogen tetroxide is splashed on the skin or in the eyes, immediate removal of the liquid is essential to avert serious injury. If splashed into the eyes, flush with large amounts of water for at least 15 minutes, with a companion assisting by holding the eyes open if necessary. Medical assistance should be summoned immediately; however, if it is necessary to choose between flushing the eyes and summoning a physician, the eye washing should take precedence for the first 10 minutes, after which medical assistance is summoned and then the eye washing resumed. Administration of anything else, including neutralizing agents, should be done only at the direction of a physician. Splashing of the liquid onto the skin should also be followed immediately by a thorough washing of the affected parts with large amounts of water.

Storage areas shall be well supplied with easily accessible, plainly marked deluge type safety showers for personnel, controlled by quick-opening valves capable of supplying large quantities of water under moderately high pressure.

Persons exposed to nitrogen tetroxide fumes should be removed from the contaminated area immediately. When the fumes can be seen, smelled, or sensed by the eyes or throat, there may be imminent danger. Persons exposed to the fumes should be carried and not allowed to walk: other exertion should also be discouraged. Immediately following exposure, it is difficult to determine the extent of risk involved. Persons so exposed should be under observation for at least 24 hours, despite their protestations of well-being. Those known to have been seriously exposed should be removed to a hospital. Absolute rest is essential, and patients should be kept warm but not overheated. Administration of oxygen by properly trained persons is often desirable. If breathing stops, artificial respiration (preferably with the aid of oxygen) should be applied even if medical assistance is not available. Do not administer sedatives except by direction of a physician, because of the danger of synergistic respiratory depression. Venesection may be useful to relieve an overloaded heart consequent to pulmonary edema, but because of possible complications, a physician should decide whether to institute this treatment.

3.2 Fire Hazards.

- 3.2.1 General Nitrogen tetroxide is normally stored and handled as a liquid without refrigeration. Liquid and gaseous nitrogen tetroxide are stable at ordinary temperatures. Liquid nitrogen tetroxide, by itself, will not burn, but will support combustion. Since the smoke and fumes from fires of this nature are generally toxic, fires should be approached and fought from the upwind side. Firefighters should wear full protective clothing and self-contained breathing apparatus.
- 3.2.2 Types of Fires The oxygen content of nitrogen tetroxide is about 70% by weight. When mixed with a fuel it readily supports combustion. Nitrogen tetroxide is hypergolic with UDMH, hydrazine, aniline, and furfuryl alcohol, among other fuels.
- 3.2.3 Control In case of fire, an attempt should be made to shut off the flow of either nitrogen tetroxide or fuel. Personnel should be evacuated from the contaminated area in case of spillage. If large spills of liquid

nitrogen tetroxide occur, arrangements should be made to appropriately handle or drain the liquid to a disposal system or neutralizing area. All surfaces contacted with liquid nitrogen tetroxide shall be flushed thoroughly with large quantities of water. In applying the water, care must be taken to prevent splashing or splattering. The area surrounding the fire should be thoroughly wetted down to prevent organic material such as brush and grass from igniting. Water will accelerate fuming of liquid nitrogen tetroxide. Continued application of large quantities of water will eventually dilute the oxidizer so that combustion is no longer supported. The remaining air-supported fuel fire may be extinguished by using techniques applicable to the fuel involved.

3.3 Explosion.

3.3.1 General - Nitrogen tetroxide is an oxidizer (approximately 70% oxygen); therefore, mixtures with nonhypergolic rocket fuels present an explosion hazard. Nitrogen tetroxide of commercial purity is stable at ordinary temperatures and can be safely stored in moderate-pressure vessels. (paragraph 5.3.2)

The possibility of pressure rupture of containers exists at elevated temperatures, and vapors released can form explosive mixtures with fuel vapors in confined spaces.

- 3.3.2 <u>Prevention</u>- Nitrogen tetroxide must be stored and handled in well-ventilated spaces, remote from fuels. Storage areas should be maintained at moderate temperatures.
- 4. SAFETY MEASURES.
- 4.1 General.

All operations involving the handling of nitrogen tetroxide, shall be performed by groups of two or more persons.

4.2 Education of Personnel.

The following subjects should be explained thoroughly to all persons working with the storage, handling, or transfer of nitrogen tetroxide:

- a. Nature and properties of nitrogen tetroxide in both the liquid and vapor states. Emphasis must be given to the high toxicity and relatively high volatility of nitrogen tetroxide.
- b. Compatible materials of construction.
- c. The proper equipment and its operation.
- d. Use and care of personal protective equipment and clothing.
- e. Safety measures, self-aid, and first aid.

Trained supervision shall be provided for all hazardous activities involving nitrogen tetroxide.

4.3 Personal Protection.

The principal personal hazards associated with the handling of nitrogen tetroxide are:

- a. Exposure of the skin to the liquid or high vapor concentration.
- b. Inhalation of the vapors.
- 4.3.1 Hand and Foot Protection The hands and feet are always subject to liquid contamination during the handling of liquid propellants or propellant equipment. Gloves and footwear which do not let nitrogen tetroxide through to the skin must be worn at all times.

The gloves used must protect the hands from nitrogen tetroxide and allow free movement of the fingers. A vinyl-coated glove which meets these requirements is the Type R-1, Specification MIL-G-4244.

Protective footwear must be worn over regular safety footwear and it must be high enough to fit under the protective trousers. Footwear made of the approved materials (paragraph 5.2.3) is not available commercially, but it may be made of natural, reclaimed or GR-S rubber and used with reasonable safety if any contamination is washed off quickly. Frequent inspections should be made of the boots to detect flaws which might result in personal injury.

Head, Face, and Body Protection - Rocket fuel handler's coveralls made of vinyl-coated glass cloth have been found suitable for use with nitrogen tetroxide. Polyethylene clothing may also be worn. Fiberglass clothing impregnated with acid-resisting plastic such as Teflon or Kel-F is excellent for handling nitrogen tetroxide. Rubber may burn in prolonged contact with nitrogen tetroxide. The clothing must cover all parts of the body subject to exposure. Each clothing item must be adjusted so as to prevent body contamination, or drainage into footwear or gloves from leaks or spills.

Specification MIL-C-12527, Rocket Fuel Handler's Coverall, covers a one-piece vinyl-coated glass cloth coverall which has been found suitable for use with nitro tetroxide.

An approved type hood must be worn for head protection. Specification MIL-C-12525, Rocket Fuel Handler's Hood, covers a vinyl-coated glass cloth hood with plastic visor which has been found suitable for use with nitrogen tetroxide.

The wearing of plastic-coated coveralls and hood may become quite uncomfortable under high temperature and humidity. In order to alleviate the discomfort to the operator and prevent overheating and possible heat prostration a cooling coverall may be worn over the protective coverall. This outer garment is made of knitted cotton yarn and is kept soaked with water to get maximum evaporation for cooling.

Specification MIL-C-12528, Rocket Fuel Handler's Cooling Coveralls, covers a one-piece circular knit cotton yarn coverall available in white or olive green.

A similar covering which also cools by water evaporation is available for the hood. Specification MIL-H-12529, Outer Water Evaporation Hood, covers a hood made of terry cloth and is available in olive green.

4.3.3 Respiratory Protection - Whenever the fumes from nitrogen tetroxide can be seen, and/or when they exceed the threshold limit, respiratory protection approved for use with nitrogen tetroxide shall be worn.

Various types of protection and their limitations are noted below:

a. Self-Contained Breathing Apparatus - This type provides the most reliable respiratory protection against gases or mists.

Suggested Source:

Scott Air-Pak All-Purpose Self-Contained Breathing Apparatus
Scott Aviation Corp., Lancaster, New York.

M. S. A. Air Mask with 40 cu ft capacity air cylinder. Bureau of Mines approval 1310.
Mine Safety Appliances Co., Pittsburgh, Pa.

b. Canister Type Masks - The use of this type of mask is of limited value in providing respiratory protection against nitrogen oxides. It must not be used in an atmosphere containing large volumes and high concentrations of gasses or mists, or in enclosed areas where there is no natural or forced-draft ventilation. The U.S. Army Chemical Corps Rocket Propellant Gas Mask, M21, Cm1C, Purchase Description No. 197-54-70, has been approved for use with nitrogen tetroxide.

Suggested Source:

Willson Industrial Gas Mask with Canister LG6G for use with fuels and oxidizers. Willson Products Division, Reading, Pa.

Willson Industrial Gas Mask with Canister LG3 for use with nitrogen oxides--Bureau of Mines approval number 1450.

Willson Products Division, Reading, Pa.

All Service Gas Mask, Model S, Bureau of Mines approval number 1434, Mine Safety Appliances Company, Pittsburgh, Pa.

Industrial Gas Mask Canister, Type GMB, Mine Safety Appliances Company, Pittsburgh, Pa.

c. Military Type Masks - Although some measure of protection is afforded, the adequacy of the military type gas mask is influenced by both the concentration of the fumes and the duration of the exposure. Data relative to the protection afforded by this type of mask is quite limited, therefore the degree of protection against nitrogen tetroxide cannot be stated in exact terms. (Ref. 6)

5. TRANSFER AND HANDLING.

5.1 General.

Nitrogen tetroxide of commercial purity is stable at ordinary temperatures; it exhibits only the reversible dissociation into nitrogen dioxide (Ref. 1). The vapor pressure of nitrogen tetroxide at 100°F is only 16 psig, and at 140°F it is 59.4 psig; it boils at 70°F. Therefore, refrigeration is not required to keep nitrogen tetroxide in liquid form. Nitrogen tetroxide freezes at 12°F. See paragraph 5.3.3.2 for handling under freezing conditions.

Liquid nitroger. tetroxide may be stored or transported in tank cars, or cylinders up to one ton capacity. Bulk quantities may be stored in tanks. Storage tanks for liquid nitrogen tetroxide must be of approved design, materials, and construction (paragraph 5.3.2.1) and must be suitabily housed.

Normal safety precautions usually taken for handling any toxic material should be strictly observed. Storage, transfer, and test areas must be kept clean, and free from trash, fuels, and combustibles. These areas must be inspected frequently. In general, the precautions taken for handling liquid nitrogen tetroxide are very similar to those for furning nitric acid (Ref. 2).

5.2 Materials.

5.2.1 <u>General</u> - Any organic material that may be readily oxidized is unsatisfactory for use in the handling and storage of liquid nitrogen tetroxide.

Materials listed in this section have been tested and found to be satisfactory for general use in the handling and storage of liquid nitrogen tetroxide. However, this list is not intended to exclude other materials found by test to be as good. Other materials should be tested prior to use.

5.2.2 Metals - Although nitrogen tetroxide is not corrosive to most common metals, selection of a metal should be governed by the moisture content of the nitrogen tetroxide. Listed below are metals that are satisfactory for the service indicated.

For service when moisture is 0.1% or less:

- a. Carbon steels.
- b. Aluminum.
- c. Stainless Steels.
- d. Nickel.
- e. Inconel.

For service under wet conditions (more than 0.1% of moisture):

a. Stainless steel (300 series).

Listed below are metals that are unsatisfactory:

- a. Brass.
- b. Bronze.
- c. Cadmium.
- d. Copper.
- e. Lead.
- f. Magnesium.
- g. Silver.
- h. Titanium.
- i. Zinc.
- Non-metals Because nitrogen tetroxide is very reactive with most organic compounds, the nonmetallic materials with which it is compatible are very few in number. The following is a list of non-metals that may be used for specific applications:
 - a. Ceramic.
 - b. Pyrex Glass.
 - c. Teflon.
 - d. Kel-F (unplasticized).
 - e. Asbestos (cotton free).
 - f. Polyethylene (limited use).

Ceramic and pyrex glass are limited because of their fragile nature.

- 5.2.4 <u>Lubricants</u> Hydrocarbon lubricants react with oxidizers and must be avoided. The following is a list of lubricants which are inert to strong oxidizers and may be used:
 - a. Fluorolube series.
 - b. Nordcoseal--147 and DC 234S.
 - c. Kel-F Polymer Oil.
- 5.3 Liquid Handling Equipment.
- 5.3.1 General Nitrogen tetroxide may be stored in either stationary or mobile tanks or cylinders of approved design, materials, and construction.

Equipment should be delivered without lubricants, packings, or gaskets, thereby avoiding the manufacturer's possibility of installing a prohibited material. The approved materials may then be installed, thus eliminating a safety hazard.

When equipment has a coating of grease for protection during shipment, this protective coating must be removed and the equipment thoroughly cleaned before installation.

All exposed screws, bolts, nuts, and washers used to assemble and secure equipment used in nitrogen tetroxide service must be made of stainless steel.

- 5.3.2 Containers Local requirements will determine the size and number of storage vessels. It is recommended that more than one tank be used at an installation where shipment of nitrogen tetroxide will be received in railroad tank cars or mobile tankers. This type of facility is desirable so as not to delay the unloading of commercial carriers. Nitrogen tetroxide is neither shipped nor stored in drums.
- 5.3,2.1 Tanks Tanks of 11,000-gallon capacity are most acceptable for permanent storage as nitrogen tetroxide deliveries by tank cars are approximately 8,000 to 10,000 gallons, making it mandatory that sufficient space be available to receive the shipment. Horizontal tanks mounted on reinforced concrete saddles are acceptable for this service. The tanks shall be of welded construction; and they shall be designed, constructed, and tested in

accordance with the latest ASME boiler and pressure vessel code for this type of service. These tanks must be fabricated to withstand an internal pressure of at least 150 psig, and equipped with rupture discs rated at 75 psig. A sufficient number of connections, of adequate size, shall be provided on the top of the tank for connecting to the piping system and for instrumentation. A manhole of adequate size (minimum diameter 24 in.), complete with a cover plate, shall be provided on top of the tank (paragraph 7.5).

A connection may be provided in the bottom of the tank for draining and as a clean-out. A "dump valve" arrangement is recommended for this connection. It has an operating handle mounted on top of the tank to stop the flow of liquid by closing the drain connection inside the tank.

Since nitrogen tetroxide does not present a particular corrosion hazard the tanks may be equipped with bottom outlets for transfer of liquid.

5.3.2.2 <u>Cylinders</u> - Nitrogen tetroxide may be shipped and stored in several types of cylinders.

One type of cylinder is closed by a screwed plug and protective cap. The nitrogen tetroxide may be withdrawn as a gas when the cylinder is upright, or the cylinder may be inverted for the withdrawal of liquid nitrogen tetroxide. Before connecting this cylinder to the piping system, the cylinder and its contents shall be cooled to below 70°F. For cooling instructions see paragraph 5.3.3.1.

A second type of cylinder is fitted with valves for the withdrawal of either liquid or gaseous nitrogen tetroxide. This type of cylinder is made in two styles; the upright style, and the horizontal style known as a "ton container". The ton container shall be securely placed in a horizontal position with the cylinder valves in the vertical centerline. So located, gaseous nitrogen tetroxide can be drawn from the top valve and liquid nitrogen tetroxide can be drawn from the bottom valve.

Provision shall be made for the mechanical handling of the larger cylinders.

- 5.3.3 Climatic Conditions Nitrogen tetroxide may be stored or transferred in hot or cold weather if certain operating techniques are followed. In addition, the equipment must be protected from all kinds of weather. Recommendations for hot or cold weather operations are given below.
- 5.3.3.1 Hot Weather All equipment that contains nitrogen tetroxide should be protected from the direct rays of the sun. Storage tanks and transfer systems may be protected by open side, steel frame buildings with sloped roofs of corrugated asbestos. A water spray system should be installed so the tanks may be cooled occasionally to lower the vapor pressure. Under static storage conditions, elevated ambient temperatures (above the boiling point of nitrogen tetroxide, 70°F.) will cause a high vapor pressure resulting in the possibility of leaks in a closed system.

In the event a water spray system is not installed, an ordinary garden hose may be used to cool down the storage tanks or cylinders.

5.3.3.2 <u>Cold Weather</u> - Nitrogen tetroxide freezes at 12°F.; however, this will not damage the system or equipment because nitrogen tetroxide does not expand below its freezing point. If it becomes necessary to transfer nitrogen tetroxide when the ambient temperature is below 12°F., it will be necessary to heat the storage tank so the liquid can blow.

Nitrogen tetroxide storage tanks may be equipped with a jacket or external pipe coils for maintaining complete temperature control under all climatic conditions. The jacket or pipe coils should be so designed that it may be used with water as a coolant, or with steam for heating purposes.

5.3.4 Mobile Tankers - Nitrogen tetroxide may be shipped in tank trucks or semi-trailers approved by the I. C. C. for this type of service.

Specially designed refuelers and servicing semi-trailers have been used for handling liquid propellants. These vehicles are self-contained units which can perform all liquid handling operations independently of other equipment. They are equipped with one or more propellant storage tanks and pumps and a 220-volt ac gasoline-powered engine generator which supplies electric power for the pump motors if local base power is not available. Water storage tanks and pumps are also available on some models.

When connected to a prime mover the tankers may be towed over generally smooth and level hard-surfaced roads at speeds up to and including 40 mph, and over irregular terrain at speeds up to and including 12 mph.

Additional information on these vehicles may be obtained from the Commanding General, U.S. Army Chemical Center, Md., Attn: U.S. Army Cml Res and Dev Labs, Process Development Division.

- Instrumentation All instruments should be carefully selected to insure maximum accuracy and durability. Any parts of the instruments that may come in contact with nitrogen tetroxide must be made of approved materials (paragraph 5.2). Instruments equipped with a remote read-out attachment that may be mounted on a centrally located panel board are preferred. All electrical and electronic devices and switches must be enclosed in a vaportight housing. Sight glasses, or any similar instruments which may contain nitrogen tetroxide in a glass enclosure, are unacceptable due to the possibility of breakage.
- Liquid Level Indicators All storage tanks must be equipped with a liquid level indicator. It must be of adequate size and conveniently located so as to be readily visible to the operator. The gauge must be constructed so that it indicates accurately the level of the liquid and it must be leakproof under high pressure conditions. The instrument dial shall be calibrated for easy reading according to the size and capacity of the tank. The dial should have sufficient graduations clearly marked so the operator may read a measured amount without making any computations. A high and low level alarm with an automatic pump shut-off is desirable.

This section contains a description of several types of liquid level indicators available. However, selection is not limited to those mentioned below:

a. Magnetic Type. - This type operates on the principle of a float moving a magnet which in turn drives a counter and dial indicator. This type instrument may be obtained with the operating mechanism completely sealed within the tank in which case the dial chamber may be removed while the tank is under pressure.

- b. Hydraulic Type This type gauge operates on a balanced hydraulic transmission system unaffected by ambient temperature changes or the specific gravity of the material being handled. This type instrument operates under pressure and vacuum conditions.
- c. Hydrostatic Type This type instrument operates on the hydrostatic principle and is actuated by either a compressed air supply or a hand pump. It is used where the shape or construction of the tank is such that a float type gauge cannot be used.
- d. Direct Reading Type This type instrument is acceptable if the operating mechanism within the tank is completely isolated from the liquid.
- e. Ultrasonic Type This liquid level sensing system consists of a hermetically sealed probe, sensitive to the presence or absence of liquid, installed in the tank and connected by an electric circuit to a remotely located control unit. This type liquid level monitoring device must be vaportight. Its operation is not affected by foam, vapor, or droplets.
- 5.3.5.2 Pressure and Vacuum Gauges Standard type pressure and vacuum gages, of approved materials, should be used in nitrogen tetroxide service.

 The mechanism shall be protected from the liquid by a Teflon diaphragm.

 The diaphragm chamber shall contain only Fluorolube S-30 oil.
- Flow Meters A flow meter should be installed in the discharge line of the piping system to indicate the amount of liquid being delivered by the system. It must be made of approved materials. The meter should be equipped with an adjusting mechanism so an accurate measurement of the flow of liquid may be maintained. A check valve shall be installed on the outlet side of the meter to prevent reverse flow. A strainer shall be provided on the inlet side of the meter, unless a strainer is otherwise installed in the suction side of the piping system, to prevent any foreign matter from entering the meter.

This section contains a description of several types of flow meters available. However, selection is not limited to those mentioned below:

a. Volumetric Type - This type instrument operates on the positive displacement principle. The volume of fluid passing

through the meter operates pistons which in turn operate a driving gear assembly to register the amount of flow. This type meter must be installed in a horizontal line. The meter may be equipped with a "batch counter assembly" to register the amount of flow for each batch and a "total counter assembly" to register the total amount of fluid that has passed through the meter.

- b. Magnetic Type This type instrument operates on the electromagnetic induction principle as a simple ac generator in which the flowing fluid is the armature and the electrodes act as brushes. This generates an external magnetic field. The voltage produced is directly proportional to the velocity of the flowing fluid. This voltage is carried by an electric circuit to an indicator which registers the amount of liquid flow. Viscosity and density variations have no effect on flow measurement.
- c. Turbine Type This type instrument operates on the principle of fluid flow turning a rotor. As the rotor turns, it cuts through a magnetic field generated by a magnet, thus an electrical impulse is induced in a pickup coil. Since the rotor speed depends upon the liquid flow, the output frequency and voltage are proportional to liquid flow. The output signal is then fed to an instrument for actual flow indication.
- 5.3.5.4 Temperature Indicators A standard type temperature indicator should be provided for each tank. It must be made of approved materials, and constructed so that a sensitive bulb may be inserted into a thermometer well installed in the tank and connected to the indicating dial. The dial should have sufficient graduations ranging from 0°F to 130°F for accurate reading. A high and low temperature alarm is desirable.

A recording type instrument may be substituted for the one mentioned above.

Suggested Instrument Sources:

Fisher and Porter Company Hatboro, Pennsylvania

Manning, Maxwell and Moore, Inc. Stratford, Connecticut

Bowser, Incorporated Fort Wayne 2, Indiana

Rochester Manufacturing Company, Inc. Rochester 10, New York

Potter Aeronautical Corporation Union, New Jersey Acoustica Associates, Inc. Mineola, New York The Liquidometer Corporation Long Island City 1, New York

5.3.6 Pumps and Transfer Media - Transfer of liquid by pump is preferred because it considerably lessens the possibility of introducing moisture into the system. Nitrogen tetroxide may also be transferred by means of the differential pressure method, using dry compressed air (dewpoint -40°F or lower) or gaseous nitrogen.

The pump shall be constructed of approved materials and be of the positive displacement type or the centrifugal type. Positive displacement pumps, which are self-priming, are used whenever a high suction lift is required. A centrifugal pump may be used whenever a high suction lift is not required; however, this type pump needs a flooded suction, as it is not self-priming.

This section contains a description of several types of pumps; however, selection is not limited to those listed below.

a. Rotary Type Positive Displacement Pump - This type pump is self-priming and is constructed with sliding vanes which are self-adjusting. A mechanical seal must be used to prevent leakage around the drive shaft. A pressure relief valve must be used with this type of pump. It must be set to relieve at 5 to 10 pounds higher than the operating pressure and be capable of relieving the entire capacity of the pump at full rated flow. The relief valve must be connected to the piping system so that it discharges back into the storage tank. This type pump has given satisfactory service in the handling of furning nitric acid.

Suggested Source:

Blackmere Pump Co. Grand Rapids 9, Michigan

b. Centrifugal Type Pump - A magnetically driven seal-less centrifugal pumping unit in which the pump and motor are built into a single integral unit may be used. This pump has only one moving part, a combined rotor and impeller assembly, which is driven by the rotating magnetic field of an

induction motor. The pumped liquid is isolated from the motor windings.

Suggested Source:

Chempump Corp.
Philadelphia 18, Pennsylvania

A cartridge type strainer should be used with all pumps. The strainer should be connected directly to the suction port of the pump to prevent any foreign matter from entering and damaging the internal working parts.

5.3.7 Valves.

5.3.7.1 Control Valves - Ball type valves with the pipe ends welded to the piping are preferred. The valve body and all internal parts must be made of approved materials. Valves shall be identified by a numbered tag as shown on Flow Diagram (paragraph 7.4). The number shall be large enough so as to be easily readable and it must be securely attached to the valve. All valves not easily reached for hand operation must be equipped with an extension handle.

Stainless steel needle valves should be used in nitrogen service.

- 5.3.7.2 <u>Pressure Relief Valves</u> Liquid pressure relief valves and vapor pressure relief valves, of approved materials, shall be installed throughout the system where applicable. They shall operate automatically.
 - a. Liquid Pressure Relief Valves A liquid pressure relief valve shall be installed in conjunction with each positive displacement pump and should be set to relieve at 5 to 10 pounds higher than normal working pressure and capable of relieving the entire capacity of the pump at full rated flow. This relief valve must be connected to the piping system so that it discharges the liquid into a storage tank.
 - b. Vapor Pressure Relief Valves A vapor pressure relief valve shall be installed on the top of each tank and should be set to relieve at 75 psig. This relief valve must be connected to the piping system so that it discharges the vapors into the fume absorption system.

When venting to the atmosphere it must be done through a stainless steel (type 304) vent stack, located away from the working area and discharging at least 50 feet above the highest operating level.

5.3.8 Pipe, Fittings, and Swing Joints - The pipes, fittings, and swing joints shall be of approved materials and rated at 150 psig minimum working pressure. The installation of piping, fittings, and swing joints shall be accomplished by welding whenever possible; however, if it is necessary to use threaded connections, teflon tape thread sealant (T-film) is preferred.

As an alternate, a thread sealing compound of water glass (disodium silicate) and graphite has been used successfully.

Flanged connections should be used in joining the piping system to all pieces of equipment. A sufficient number of flanged connections should be placed throughout the piping system to facilitate dismantling.

Flexible connections, swing joints, and hoses should be used to connect the piping system to any mobile equipment.

Identification of piping systems shall be in accordance with MIL-STD-101. (The primary warning color shall be brown. The secondary warning arrow color shall be black.)

- 5.3.9 Hoses Hoses shall be made of approved materials and designed for high suction lift service. Liners for hoses should be made of pure teflon or stainless steel as other materials have become hard and brittle after exposure to nitrogen tetroxide.
- 5.3.10 Gaskets and O-Rings Gaskets and O-rings fabricated from any of the applicable approved non-metals listed under Section 5.2.3 may be used.
- 5.4 Electrical Equipment.
- 5.4.1 General The careful selection of electrical equipment is an important factor in the safe and efficient handling of nitrogen tetroxide. Vaportight electrical equipment, so enclosed that vapor will not enter the enclosure, must be used to prevent internal damage from the corrosive vapors of N₂O₄.

This type of equipment must be used in hazardous locations where N₂O₄ is used in conjunction with fuels which present a fire or explosion hazard.

This section contains information on electrical motors, controls, and accessories recommended for use with nitrogen tetroxide.

5.4.2 <u>National Code Requirements.</u> The National Electrical Code is now widely accepted as giving the minimum requirements for electrical equipment, and for the installation of electrical equipment to safeguard persons and property from electrical hazards.

Electrical equipment used in liquid propellant handling and storage areas, and the installation of it, shall comply with the requirements of the latest edition of the National Electrical Code as a minimum. In certain cases, such as those occupancies involving explosive mixtures, it may be necessary to exceed the requirements of the code, in order that electrical installations will not create an undue hazard.

Vaportight electrical equipment listed by the Underwriters'
Laboratories for use in hazardous locations or approved by other recognized testing agencies is acceptable when used for the conditions of operation intended (ref. 3).

Motors, Controls, and Accessories. Motors, starters, switches, receptacles, etc., are subject to severe damage from corrosion if exposed to nitrogen tetroxide vapors. Vaportight electrical equipment must be used to prevent internal damage and subsequent failure. (Vaportight means that vapor will not enter the enclosure which houses the mechanism.) Electrical equipment and wiring methods listed by the Underwriters' Laboratories for use in Class I, Group D locations have proved satisfactory for use under heavy concentrations of N2O₄ vapors. Proper grounding procedures must be followed to prevent a possible shock hazard.

Hermetically sealed aircraft type switches and relays have been used on liquid propellant installations where space limitations prohibit the use of conventional vaportight apparatus. This equipment offers protection from corrosion and humidity. The requirements for relays of this type are outlined in MIL-R-6106, General Specification for Aircraft Electric Relays.

Suggested Sources:

Crouse-Hinds
Syracuse, New York

Cutler-Hammer Milwaukee 1, Wisconsin

- 5.5 Fume and Liquid Decontamination.
- 5.5.1 General This section contains information on the various techniques and methods used to control or reduce the hazards resulting from spills or leaks of nitrogen tetroxide fumes or liquid.

Since the odor of nitrogen tetroxide cannot be relied upon to indicate toxic concentrations, monitoring devices should be employed in work areas to warn of leaks (paragraph 6.2). In the event of contamination by spills, leaks, or fires immediately evacuate exposed or affected personnel from the contaminated area and render treatment as necessary. Allow only personnel protected by approved face, body, and respiratory protective equipment to enter or remain in the area.

Provision should be made for readily approaching all control valves, and valves used in isolating various parts of the storage area to prevent the nitrogen tetroxide from blanketing the entire area in the event of leakage.

5.5.2 <u>Spills and Leaks</u> - If the proper drainage system and neutralizing facilities are installed (paragraph 7.12 and 7.15), it is necessary only to flush the spilled liquid down the drain. If these facilities are not available, disposal of nitrogen tetroxide may become a problem.

In the event of a large "pool" spill, the liquid may be pumped into suitable containers and transferred to an approved disposal area. Flush all contaminated surfaces with large quantities of water, making sure that all washings are directed or drained to that area. Ventilate the contaminated areas.

Wash contaminated surfaces (other than aluminum) from which gross contamination has been removed, as noted above, with sodium bicarbonate or sodium carbonate water solution and rinse with water as a final step

in neutralization. The nitrogen tetroxide may be disposed of by burning, or neutralization and dilution, provided the acid level is retained below acceptable values in the ultimate disposal area.

Small leaks may be taken care of temporarily by a continuous flushing with water at the source of the leak. In elevated temperatures, some leaks may be reduced by cooling the container with a water spray, or periodic water applications with a hose.

In some cases, it may not be advisable to flush with water, but instead allow the vapors to evaporate. Flushing with water will increase the NO₂ vapors considerably. Nitrogen tetroxide is only slightly soluble in water, and nitrogen tetroxide is heavier, remaining at the bottom and slowly converting to nitric acid. Disturbing this formation may result in the release of additional noxious fumes.

5.5.3 Fume Decontamination - Every effort must be made to provide the maximum protection for operating personnel from the hazards of toxic vapors resulting from the transfer and handling of nitrogen tetroxide. In some cases, the fumes may be vented slowly from elevated stacks when atmospheric conditions are favorable. However, for safe and positive venting of noxious fumes under all circumstances, a fume absorption system must be used. This system neutralizes the fumes liberated during the transfer, storage, or dispensing of nitrogen tetroxide and absorbs the fumes resulting from normal handling and disposal operations.

Complete information on the design and operation of a fume absorption system is included in this manual under paragraph 7.14.

5.5.3.1 Fog Nozzles - The use of water spray fog nozzles has been found to be very effective in controlling the fumes from nitrogen tetroxide spills.

The nozzles are located around the periphery of each transfer and/or handling station at ground level, and pointed upward. With this arrangement, it is possible to contain the fumes in the spill area, and drive them to the center of the fog pattern where they are diluted and dispersed well above the ground. A second set of nozzles installed at least six

feet above ground level nozzles increases the effectiveness of the dispersal.

Information given here is based on tests conducted at the U.S. Army Chemical Center, Md. In this test installation, 1-inch nozzles were spaced about 6 feet apart, connected by a header to the water supply. A booster pump was used to maintain a water pressure of 45 psig. Details of these tests are contained in a report (Ref. 9).

Disposal of Liquids - A disposal system (paragraph 7.12) and a neutralization system (paragraph 7.15) should be installed for disposing of large quantities of nitrogen tetroxide. If these facilities are not available, an alternate method, which is quite time-consuming, is to drain or pump nitrogen tetroxide into a basin or pond where it can be neutralized with soda ash, or allowed to boil off--provided the area is unpopulated. Neutralization should take place before dumping into a sewer or waterway (Ref. 5).

Another method of disposal of large quantities of nitrogen tetroxide is by burning with a fuel such as kerosene or liquid petroleum gas.

5.5.5 Decontamination of Equipment - Before removing pipes, fittings, valves, or other components from a piping system, make sure that the system has been thoroughly drained, and flushed with large quantities of water. Equipment items such as pumps or flow indicators having pockets or crevices which may contain small quantities of concealed liquid shall be thoroughly inspected and recleaned if necessary before sending to the shop or storage area.

Care must be taken to remove any grease, lubricants, or compounds from all internal (and external) surfaces of piping components before installing in the system. After the system has been reassembled, it must first be purged with methylene chloride to further remove any traces of organic matter. The system is then drained and throroughly dried.

A new system must also be given the methylene chloride--dry air treatment described above.

6. OPERATING TECHNIQUES AND PRECAUTIONS.

6.1 General.

This section contains information on equipment and operating techniques pertaining to safety measures and procedures. Basically, a properly engineered system is essential for safe and efficient handling of nitrogen tetroxide. Compatible materials only will be used, otherwise injury or even death may result.

All operations involving the transfer of nitrogen tetroxide should be performed out-of-doors. In the event that natural ventilation is not possible or adequate, downdraft ventilation should be provided.

Good housekeeping must be maintained at all times since nitrogen tetroxide reacts readily with organic matter. Working areas shall be kept clean and free of any materials other than those recommended.

6.2 Fume Detection.

Nitrogen tetroxide volatilizes readily, giving off yellowish to reddish-brown fumes containing a mixture of nitrogen tetroxide (N₂O₄) and nitrogen dioxide (NO₂). A detection instrument should be provided as a means of controlling these hazards in propellant operations through quick testing of suspected atmospheres. Nitrogen dioxide requires especially rapid and reliable determination for maximum protection since this gas has a faint odor and irritates only slightly even in dangerous concentrations.

There are several types of instruments available to warn operating personnel when the concentrations of nitrogen tetroxide in the air reaches the MAC. These instruments range from field detection kits to continuously operating systems complete with alarms.

a. Nitrogen Dioxide Detector Kit - A portable field kit containing reagent bottles, sampling syringes, and color plaques for determining oxides of nitrogen in the atmosphere. Range is 1 to 500 ppm of the contaminant in air.

Suggested Source:

Catalog No. DZ-4747Z Mine Safety Appliances Company Pittsburgh 8, Pennsylvania

- b. Automatic Nitrogen Dioxide Alarm, E24R2 A detection instrument which continuously measures the conductivity of water which has been exposed to air drawn through a gas inlet. Absorption of nitrogen dioxide causes a proportionate increase in conductivity and this increase is shown on a meter mounted on the face of the alarm. A concentration exceeding 10 ppm of nitrogen dioxide in air will cause a light and a buzzer to be actuated.
- U. S. Army Chemical Corps Purchase Description No. 197-54-631.
- U. S. Army Chemical Corps Operating Directive, DIR-CWL-270C.

Suggested Source:

Davis Engineering Company Newark, New Jersey

Various other types of instruments are available, some of which employ coulometric titration cells.

Suggested Sources:

Western Dynamics, Inc. Los Angeles, Calif.

Micro-Path, Inc. Los Angeles 45, Calif.

Union Industrial Equipment Corporation Port Chester, New York

6.3 Hazardous Vapor Travel.

It may be desirable to predict downwind travel of a toxic vapor from an accidental release of a propellant. These predictions are based on calculations which can be performed by several means. One convenient method involves the use of "Downwind Vapor Hazard Nomographs," also available in the form of a slide rule.

These nomographs can be useful in planning the location and dispersion of buildings and personnel in the design of a facility. They may also be used in prior planning to facilitate decisions in emergency conditions.

Additional information on these nomographs may be obtained from the office of the Chief Chemical Officer, Department of the Army, Washington 25, D. C.

6.4 Installation Precautions.

All nitrogen tetroxide tanks shall be provided with a vapor pressure relief valve of adequate size, set at a safe working pressure which is determined by the design of and materials used on the tank. Rupture discs are also used and must be selected to suit the individual application. Adequate vent lines discharging into a fume scrubbing system shall be provided when the facility is located in areas which prevent venting to the atmosphere. These vent lines are installed on the outlet of the pressure relief equipment. When venting to the atmosphere, it must be done through a stainless steel (type 304) vent "stack" located away from the working area and discharging at least 50 feet away from the highest operating level.

All packing gland seals around pump shafts, valves, etc, shall be equipped with polyethylene shields to prevent nitrogen tetroxide from spraying directly on operators in case of failure.

6.5 Water Supply.

Adequate water shall be provided for decontamination and flushing purposes, deluge systems, showers, and eye baths. A water flow of at least 50 gallons per minute must be available for flushing. One or more water hoses with quick-opening valves must be available at all times.

If fog nozzles are used for dissipation of nitrogen tetroxide fumes, the local water supply may have to be supplemented by a pump to maintain the pressure necessary for efficient fume dispersal.

6.6 Operating Precautions.

In all operations involving the handling of nitrogen tetroxide, the operating personnel shall wear approved protective clothing, footwear, and respiratory equipment. There shall never be less than two operators on any operation at any time.

Before charging any system with nitrogen tetroxide, it is of utmost importance that the storage tank, pipe lines, pumps, valves, and fittings are flushed free of oil, other organic materials, scale, or foreign matter.

Methylene chloride is suitable for flushing purposes since it is compatible with and soluble in nitrogen tetroxide. If the storage tank or any part of the piping system contains moist air, it should be purged thoroughly with dry compressed air or nitrogen before charging is begun. Caps should be installed on free ends of flexible hose to prevent entrance of moist air when the hose is not in use.

6.7 Quantity Distance Tables.

No acceptable quantity distance tables exist for liquid propellants (Ref. 3).

7. TYPICAL BULK STORAGE FACILITY.

7.1 General.

The purpose of this section is to provide plans and specifications for a typical installation for the storage and handling of nitrogen tetroxide. Provision has been made for receiving the nitrogen tetroxide in tank car, tank truck, or cylinders and for dispensing the liquid through a closed system to the servicing vehicles.

A set of drawings accompanies the text, giving necessary information on the design, construction, installation, and operation of the facility. Flow diagrams of the various piping systems are included for use with the step-by-step instructions provided in the manual.

Local requirements will determine the amount of storage required.

The 11,000-gallon tank units are recommended since nitrogen tetroxide

deliveries may be made by tank cars having a capacity of 8,000 to 10,000 gallons, thus insuring that space is available for a full tank car. Although the installation may require only one storage tank, it is recommended that at least two tanks be provided for flexibility of operation.

The information provided herein will be used only as a guide, since no attempt has been made to provide minute construction details. Construction details at each individual installation will be worked out in line with the fundamental principles contained herein. It should be stressed, however, that the principles of the piping systems should be adhered to as closely as possible. It is recommended that 2-inch pipe be used for transferring nitrogen tetroxide as indicated on the drawings, regardless of the number of tanks used. It is mandatory that the compatible materials specified be used.

Similar information for the handling of red and white furning nitric acid has been prepared and published by the Air Force (Ref. 8).

Paragraphs 7.1.1 through 7.1.9 contain general criteria. Paragraphs 7.2 through 7.16 contain more specific information, including references to drawings of each of the components of the facility. A plot plan of the entire facility is also included.

- 7.1.1 Location No acceptable quantity distance tables for liquid propellants exist at this time. It is recommended, therefore, that local base regulations be observed.
- 7.1.2 Terrain The proposed storage site should be reasonably level in order to keep grading and other earth-moving operations at a minimum. It is estimated that one 11,000-gallon tank full of nitrogen tetroxide will weigh approximately 75 tons, making it mandatory that soil characteristics be taken into consideration to provide proper footings for tank saddles, buildings, and rail facilities. Level terrain will also aid in the construction of the railroad spur. Drainage ditches should be provided where necessary to carry off the rain water.
- 7.1.3 <u>Buildings and Structures</u> No combustible materials will be used as part of the nitrogen tetroxide storage or handling structures. The purpose

of a roof over the various operating and storage areas is to provide shade and weather protection. The roofing material may be corrugated asbestos. Conventional lightning protection will be installed. There shall be no expansion joints in the concrete drain basins. All construction joints are to be sealed with cement mortar

7.1.4 Roadways - The various operating units in the handling and storage area are connected by a series of roadways within the fenced-in area (see Dwg. F142-2-8).

Adequate heavy-duty roadways are required in the area, since the maximum tank truck load may be about 40 tons. The roadways shall be made of concrete. The nitrogen tetroxide may be delivered in tank trucks or ton containers.

- 7.1.5 Railroad Sufficient trackage on the rail spur should be available to accommodate four tank cars simultaneously. Installation of a car spotter is recommended.
- 7.1.6 Water Supply The storage area shall be well supplied with easily accessible, plainly marked deluge-type safety showers for personnel, controlled by quick-opening valves capable of supplying large quantities of water under moderately high pressure (about 50 gpm). Cold water eye baths should also be provided at all operating stations. Hose connections are also necessary for flushing purposes and for cooling the equipment. If fog nozzles are installed for control of nitrogen tetroxide vapors, the local water supply may have to be supplemented by a pump to get the higher pressure necessary for efficient spray coverage.

In the change house, water will be required for showers and for drinking.

7.1.7 Sewage Disposal - A sanitary sewer will be required to take care of the effluent from the change house. It may also be used to carry waste from the storage facility after the waste has cleared the neutralizing system. In the event that the nearest sanitary sewer lines are too distant from the storage site, it may be necessary to construct a septic tank to accommodate the effluent from the change house.

7.1.8 <u>Electrical</u> - Electric power must be provided for the pumps, lights, instruments, laboratory equipment, etc, in the area. All electric lines will be installed in rigid metal conduit. All electrical equipment subject to exposure to nitrogen tetroxide fumes must be vaportight to prevent corrosion and failure. Weatherproof lighting and floodlights will be installed at all working areas.

A master switch to shut off all power in the area (other than lights) in the event of an emergency should be located on the outside of the change house on the wall nearest the storage area (paragraph 7.11).

Steel utility poles should be used.

7.1.9 Nitrogen Supply - Nitrogen gas shall be supplied to the storage and transfer system, the disposal system, and the container storage area. If the nitrogen tetroxide handling and transfer facility is located near an installation having a nitrogen supply, that nitrogen source will be acceptable. However, if the facility is located in a remote area, provision must be made for storing nitrogen in bulk lots. Nitrogen is usually shipped in bottles, containers, or mobile tank trailers. The above-mentioned shipping methods will dictate the area or place for storing the nitrogen gas. Stainless steel needle valves and tubing will be used to connect the nitrogen gas to the various components.

7.2 Bulk Storage Facility - Plot Plan. (Dwg. F142-2-8)

This drawing shows a plot plan of a typical "two tank" installation with all the necessary component units for a complete handling and storage facility. The fundamental component of the facility is the storage and transfer system, wherein the basic transfer operations are performed. The storage tanks and pumps of this system are connected to the railroad tank car unloading platform, a mobile tanker station, and a container storage installation. Noxious fumes emanating from this system are piped to a fume absorption system, where the vapors are "scrubbed" before release to the atmosphere. A disposal system is provided for treating large or small quantities of nitrogen tetroxide before transferring them to the neutralizing system. A neutralizing system is provided for diluting and treating any waste liquids or

acids prior to discharge. Noxious fumes from the disposal system and the neutralizing system are piped to a separate fume absorption system.

NOTE: If the facility is situated in a remote area, it may not be necessary to install a fume absorption system. The nitrogen dioxide fumes may be vented directly to the atmosphere if there is no danger to personnel. The vent should discharge the fumes at least 50 ft. above the normal working level.

The change house provides dressing rooms, locker space, and office and laboratory facilities.

The design is not to be construed as binding, as local regulations and the actual physical geography of the area will dictate the location of the various units of the installation. The facility should be installed in a depression in the ground made by excavating the earth. This will serve as a "dyke". It should be large enough to contain all the liquid that can be held in all of the storage vessels at any one time, plus 10%. This is necessary to confine all of the liquid in the event of complete destruction of the facility. The entire area is enclosed by an 8-foot galvanized chain link fence with three strands of barbed wire along the top. Entrance gates are provided where necessary. The fence should be inspected and maintained at regular intervals. No combustible materials will be used for constructing any part of the facility.

7.3 Storage and Transfer System. (Dwg. E142-2-9)

This drawing shows a detailed piping arrangement for connecting two 11,000-gallon storage tanks to a railroad tank car unloading station and a service vehicle refueling station. The piping system is also connected to a container storage area, a fume absorption system, a liquid disposal unit, and a neutralizing system. The two tanks are manifolded together and connected to two positive displacement pumps. This arrangement allows for all necessary transfer operations. Nitrogen gas is provided in the event transfer of the liquid by the differential pressure method is desired. All the necessary valves and instrumentation are provided.

During any transfer operation the fume absorption system shall be operated as a "falling film" scrubber. See paragraph 7.14.5.4.1.b.

When the storage tanks are used under normal storage conditions, the fume absorption system shall be operated as a "static" scrubber. See paragraph 7.14.5.4.1.a.

7.4 Storage and Transfer System - Flow Diagram. (Dwg. D142-2-10)

This flow diagram identifies all manually operated valves by a number. All pipe lines and other equipment are identified by name. The flow diagram should be studied carefully, and all operators should have a thorough understanding of which valves must be operated to perform a particular transfer operation. Copies of this flow diagram should be posted at several conspicuous places. The reproduction (signs) shall be easily readable, durable, and they must be protected to prevent disfiguration or damage.

- when not in use, all manually operated valves must be closed, and the two transfer pump operating switches locked. The operators must be thoroughly informed of the hazards present when handling nitrogen tetroxide. It is mandatory that operating personnel be completely familiar with the operation of the system and the reasons for the many functions. The operators should be thoroughly familiar with all the equipment and be able to perform minor repairs and maintain the equipment in good operating condition. The operators will also be required to know how the fume absorption system and the liquid disposal and neutralizing systems operate. The operators must know how to take care of any unusual spillage or emergency that may occur.
- 7.4.2 Operating Instructions Before any operation is started, the operator shall make sure the entire facility is ready to function and the nitrogen tetroxide piping system is clean and dry. See paragraph 6.6. The following are step-by-step instructions for several typical transfer operations:
 - a. To transfer Nitrogen Tetroxide from Tank Car to Storage Tank A Using Pump 1 (Vent to Tank Car).
 - (1) Spot the tank car so that it is centered over the drainage basin and the dome of the tank car is approximately in line with the flexible hoses.

- (2) Connect the flexible hose from the "outlet header" of the piping system to the "dip pipe" connection on the tank car.
- (3) Connect the flexible hose from the "vent header" of the piping system to the "vent" connection on the tank car.
- (4) Open the "dip pipe" valve and the "vent" valve located in the dome of the tank car.
- (5) Open valves 17 and 19. This will balance the pressures between the two pieces of equipment.
- (6) Open valves 3, 5, 9, and 10.
- (7) Start the pump.
- NOTE: During any transfer operation, one operator shall carefully observe all instruments on the panel board to make sure the equipment is operating properly. If any emergency occurs during the transfer operation, all equipment must be shut down immediately and the trouble corrected. If necessary, the fog nozzle spray system shall be activated to control any escaping vapors and the necessary emergency measures taken to flush away any liquid spills.
 - (8) When the desired amount of liquid has been transferred, turn off the pump. The operator can determine when the proper amount of liquid has been transferred by reading either the liquid level indicator or the flow meter.
 - (9) Close valves 3, 5, 9, 10, 17, and 19.
 - (10) Close the "dip pipe" valve and the "vent" valve located in the dome of the tank car.
 - (11) Disconnect the hoses from the tank car. Care must be exercised performing this function as N₂O₄ liquid and vapors are present in the hose lines. These hoses must be "capped" immediately.
- b. To Perform the Same Transfer Operation, but Vent Storage
 Tank A to Fume Absorption System, Proceed as Outlined in
 "a" Above, Except:
 - (1) Open valves 17 and 21 to release any excess pressure which may be in the tank car.
 - (2) Close valve 17.

- (3) Connect the flexible hose from the "nitrogen header" of the piping system to the tank car. Open the valve in the dome of the tank car for this connection.
- (4) Vent storage tank A by opening valve 19.
- (5) Open valve 24. Regulate the flow of nitrogen as required.
- NOTE: If there is no fume absorption system in the facility, the fumes are vented directly to the atmosphere by using valve 29. This procedure may be used only if venting the fumes to the atmosphere does not create a hazard to personnel.
- c. To Perform Either Transfer Operation as Stated Above, but Using Pump 2, Proceed as Outlined in "a" or "b" Above, Except:
 - (1) Open valves 13 and 14 instead of valves 9 and 10. Valve 22 must be opened for this operation to balance the liquid in the two storage tanks because the pressure relief valve on pump 2 discharges into tank B, which may contain liquid N₂O₄.
- d. To Transfer Nitrogen Tetroxide from Tank Car to Storage Tank A, Using Nitrogen Pressure.
 - (1) Same as instruction "a" 1.
 - (2) Same as instruction "a" 2.
 - (3) Connect flexible hose from "nitrogen header" of piping system to tank car.
 - (4) Open the "dip pipe" valve and the "nitrogen connection" valve located in the dome of the tank car.
 - (5) Open valves 19 and 21.
 - (6) Open valves 3, 5, and 15.
 - (7) Open valve 24. Regulate the flow of nitrogen as required.
 - (8) When the desired amount of liquid has been transferred, close all valves.
 - (9) Disconnect flexible hoses from tank car.

The transfer of nitrogen tetroxide from any one piece of equipment to any other piece of equipment may be accomplished as described above, using the appropriate valves as shown on the flow diagram.

7.4.2.1 System Capability - The capabilities of the system are by no means limited to the operations described in a., b., c., and d., above.

The piping arrangement is designed for maximum flexibility, thereby permitting a variety of transfer operations to meet every conceivable condition. Instructions for all transfer operations are given in the Tables I through V.

7.5 Storage Tank. (Dwg. E142-2-11)

This drawing shows details of a typical 11,000-gallon storage tank. The tank is also used for holding and treating nitrogen tetroxide in the liquid disposal system. It is made of stainless steel with all-welded construction. The tank is designed to withstand an internal pressure of 150 psig (min.) and conforms to the latest ASME boiler and pressure vessel code. The tank is equipped with a rupture disc rated at 75 psig. The tank has a 24-inch diameter manhole and eleven nozzle connections on the top for connecting to the piping system and instrumentation. Two nozzle connections are provided on the bottom. A nozzle connection is provided on the manhole cover for attaching a sampling device. A "dump valve", used to close a bottom outlet from inside the tank in an emergency, is shown. It may be installed on the tank, if desired.

The tank may be equipped with a jacket or external pipe coils for maintaining complete temperature control under all climatic conditions. The jacket, or pipe coils, should be so designed that it may be used with water as a coolant or with steam for heating purposes.

7.6 Storage Tank Installation. (Dwg. E142-2-12)

This drawing shows a typical "two-tank installation" for storing nitrogen tetroxide. It shows two 11,000-gallon tanks mounted on concrete saddles in a concrete drainage basin. A steel framed "open side" building with a corrugated asbestos roof, for weather protection, is provided. The roof is made with an opening the entire length of the building to allow any fumes to escape. The opening has a cover to prevent rain and direct rays of the sun from entering. A water-fog spray-piping system is installed around the periphery of the drainage basin to control and dissipate any nitrogen tetroxide vapors. It is also used to flush away any spills or for cooling the tanks. A similar system is installed on the roof to control the fumes that will escape to the atmosphere through the ventilator in the roof.

TABLE I
TRANSFER OPERATIONS FROM TANK CAR

		TO		METHOD	VENT TO	OPEN VALVES
1	Storage	Tank	"A"	Pump 1	Tank Car	3, 5, 9, 10, 17, 19
2	11	11	11	111	Scrubber	3, 5, 9, 10, 19, 21, 24
3	**	11	11	Pump 2	Tank Car	3, 5, 13, 14, 17, 19, 22
4	11	11	11	H	Scrubber	3, 5, 13, 14, 19, 21, 22, 24
5	11	11	11	Pressure	11	3, 5, 15, 19, 21, 24
6	Storage	Tank	"B"	Pump 2	Tank Car	4, 5, 13, 14, 17, 20
7	11	*1	11	11	Scrubber	4, 5, 13, 14, 20, 21, 24
8	*1	**	11	Pump 1	Tank Car	4, 5, 9, 10, 17, 20, 22
9	11	11	"	**	Scrubber	4, 5, 9, 10, 20, 21, 22, 24
10	11	*11	11	Pressure	**	4, 5, 15, 20, 21, 24
11	Tank Ti	ruck		Pump 1	Tank Car	2, 5, 9, 10, 17, 18, 22
12	11	**		11	Scrubber	2, 5, 9, 10, 18, 21, 22, 24
13	11	11		Pump 2	Tank Car	2, 5, 13, 14, 17, 18, 22
14	H	11		11	Scrubber	2, 5, 13, 14, 18, 21, 22, 24
15	11	**		Pressure	**	2, 5, 15, 18, 21, 24
16	Disposa	.1 Unit		Pump 1		5, 9, 10, 22, 23, 24
17	11	11		Pump 2		5, 13, 14, 22, 23, 24
18	11	11		Pressure		5, 15, 23, 24
19	Recircu	late		Pump 1		1, 5, 9, 10, 22
20	11			Pump 2		1, 5, 13, 14, 22

TABLE II
TRANSFER OPERATIONS FROM STORAGE TANK "A"

	TO		METHOD	VENT TO	OPEN VALVES		
1	Tank Car				Pump 1	Storage Tank "A"	1,7,9,10,17,19
2	11	"			н	Scrubber	1,7,9,10,17,21,26
3	**	11			Pump 2	Storage Tank "A"	1, 7, 13, 14, 17, 19, 22
4	11	11			11	Scrubber	1, 7, 13, 14, 17, 21, 22, 26
5	11	"			Pressure	· ·	1,7,8,17,21,26
6	6 Storage Tank "B"				Pump 1	Storage Tank "A"	4, 7, 9, 10, 19, 20
7	**		**	11	19	Scrubber	4, 7, 9, 10, 20, 21, 26
8	11		"	**	Pump 2	Storage Tank "A"	4, 7, 13, 14, 19, 20
9	"		11	**	11	Scrubber	4, 7, 13, 14, 20, 21, 26
10	**		11	*11	Pressure	11	4,7,8,20,21,26
11	l Tank Truck				Pump 1	Storage Tank "A"	2, 7, 9, 10, 18, 19
12	11	11			11	Scrubber	2, 7, 9, 10, 18, 21, 26
13	H	11			Pump 2	Storage Tank "A"	2, 7, 13, 14, 18, 19, 22
14	11	11			स	Scrubber	2, 7, 13, 14, 18, 21, 22, 26
15	11	11			Pressure	· ·	2,7,8,18,21,26
16	6 Disposal Unit				Pump 1		7, 9, 10, 23, 26
17	**		"		Pump 2		7, 13, 14, 22, 23, 26
18	"		11		Pressure		7, 8, 23, 26
19	9 Recirculate				Pump 1		3, 7, 9, 10
20	0 "				Pump 2		3, 7, 13, 14, 22

TABLE III
TRANSFER OPERATIONS FROM STORAGE TANK "B"

		то		METHOD	VENT TO	OPEN VALVES
1	Tank (Car		Pump 2	Storage Tank "B"	1, 11, 13, 14, 17, 20
2	*1	11		••	Scrubber	1, 11, 13, 14, 17, 21, 27
3	11	11		Pump 1	Storage Tank "B"	1, 9, 10, 11, 17, 20, 22
4	11	*1		11	Scrubber	1, 9, 10, 11, 17, 21, 22, 27
5	11	11		Pressure	11	1, 11, 12, 17, 21, 27
6	6 Storage Tank "A"			Pump 2	Storage Tank "B"	3, 11, 13, 14, 19, 20
7	**	11	11	tt	Scrubber	3, 11, 13, 14, 19, 21, 27
8	11	11	11	Pump 1	Storage Tank "B"	3, 9, 10, 11, 19, 20
9	11	11	11	11	Scrubber	3, 9, 10, 11, 19, 21, 27
10	11	**	11	Pressure	11	3, 11, 12, 19, 21, 27
11	Tank T	ruck		Pump 2	Storage Tank "B"	2, 11, 13, 14, 18, 20
12	**	**		11	Scrubber	2, 11, 13, 14, 18, 21, 27
13	11	11		Pump 1	Storage Tank "B"	2, 9, 10, 11, 18, 20, 22
14	**	ŦŤ		11	Scrubber	2, 9, 10, 11, 18, 21, 22, 27
15	11	11		Pressure	п	2, 11, 12, 18, 21, 27
16	Dispos	al Unit		Pump 2		11, 13, 14, 23, 27
17	**	**		Pump 1		9, 10, 11, 22, 23, 27
18	11	11		Pressure		11, 12, 23, 27
19	Recirculate			Pump 2		4, 11, 13, 14
20	0 "			Pump 1	•••	4, 9, 10, 11, 22

TABLE IV
TRANSFER OPERATIONS FROM TANK TRUCK

	то		METHOD	VENT TO	OPEN VALVES	
1	Tank C	ar		Pump 1	Tank Truck	1,6,13,14,17,18,22
2	**	**		f1	Scrubber	1,6,13,14,17,21,22,25
3	*11	11		Pump 2	Tank Truck	1,6,9,10,17,18,22
4	*1	11		**	Scrubber	1,6,9,10
5	11	11		Pressure	11	1,6,15,17,21,25
6	Storage	Tank	"A"	Pump 1	Tank Truck	3, 6, 9, 10, 18, 19
7	11	11	11	**	Scrubber	3, 6, 9, 10, 19, 21, 25
8	**	11	**	Pump 2	Tank Truck	3, 6, 13, 14, 18, 19, 22
9	11	11	11	ti	Scrubber	3, 6, 13, 14, 19, 21, 22, 25
10	ŧi	11	**	Pressure	11	3, 6, 15, 19, 21, 25
11	Storage Tank "B"			Pump 2	Tank Truck	4,6,13,14,18,20
12	11	11	"	78	Scrubber	4, 6, 13, 14, 20, 21, 25
13	**	11	**	Pump 1	Tank Truck	4, 6, 9, 10, 18, 20, 22
14	**	11	**	Ŧŝ	Scrubber	4, 6, 9, 10, 20, 21, 22, 25
15	11	11	**	Pressure	11	4, 6, 15, 20, 21, 25
16	Disposa	al Unit		Pump 1		6, 9, 10, 22, 23, 25
17	н	11		Pump 2		6, 13, 14, 22, 23, 25
18	11	11		Pressure		6, 15, 23, 25
19	Recircu	ılate		Pump 1		2, 6, 9, 10, 22
20	11			Pump 2	••-	2, 6, 13, 14, 22

TABLE V

TRANSFER OPERATIONS FROM CONTAINER STATION

(by pressure -- vent to scrubber)

	то	OPEN VALVES				
1	Tank Car	1, 16, 17, 21, 28				
2	Storage Tank "A"	3, 16, 19, 21, 28				
3	Storage Tank "B"	4, 16, 20, 21, 28				
4	Tank Truck	2, 16, 18, 21, 28				
5	Disposal Unit	16, 23, 28				

7.7 Tank Car Unloading Platform. (Dwg. E142-2-13)

A typical railroad-type structural steel unloading platform. incorporating a counter-balanced walkway to the top of the tank car. will be constructed at the tank car unloading station. All loading and unloading will be accomplished through the dome of the tank car using flexible stainlesssteel hoses. These hoses are connected to rigid piping on the platform. Five pipe connections are provided on the platform: one for loading the tank car, one for unloading the tank car, one for connecting the tank car to the vent system, one for nitrogen gas, and one for "supplied air" to be used by the operator for respiratory equipment. All pipe connections and valves are conveniently located so the operator can easily reach them from the platform. Clamps should be provided on the platform for holding the flexible hoses when they are not in use. A stairway is provided having handrails on each side so the operator can make a hasty descent in safety, in case of an emergency, while wearing safety clothing and equipment. An emergency safety shower shall be provided on the platform. Lights shall be provided on the platform and shall conform to the requirements of electrical equipment as described in paragraph 7.1.8. The platform is made of steel and all surfaces must be finished with a protective coating suitable for use in nitrogen tetroxide service.

7.8 Drain Basin - Tank Car Unloading. (Dwg. E142-2-14)

This drawing shows a typical reinforced concrete drainage basin constructed under the railroad spur track at the tank car unloading station. It is long enough to accommodate a standard railroad tank car. The drainage basin has sufficient slope so that any liquid will flow to a drain, connected by underground pipe, to the sump. There shall be no expansion joints in the concrete. All construction joints shall be sealed with cement mortar. Rail splices over the drainage basin must be welded. No wooden ties, to support the rails, are permitted within the fenced-in area. Steel ties are recommended.

7.9 Drain Basin - Vehicle Servicing. (Dwg. F142-2-15)

This drawing shows a typical reinforced concrete drainage basin constructed in the roadway at the tank truck loading station. It is long enough

to accommodate mobile tankers and refuelers. (These vehicles weigh about 40 tons.) The drainage basin is constructed so as to confine any spilled liquid, yet allow the wheels of the vehicle to pass from the roadway onto the drainage basin. The drainage basin has sufficient slope so that any liquid flows to a drain, connected by underground pipe, to the sump. There shall be no expansion joints in the concrete. All construction joints shall be sealed with cement mortar.

7.10 Container Storage Installation. (Dwg. E142-2-16)

This drawing shows a typical storage installation for nitrogen tetroxide ton containers. It is large enough to accommodate 60 one-ton containers which are secured in position on steel rails. An overhead monorail system, complete with two electrically operated chain hoists, is installed for the mechanical handling of the containers. A reinforced concrete drainage basin is provided to confine any liquid. It has sufficient slope so that any liquid flows to a drain, connected by underground piping, to the sump. A steel-framed open-side building is included to provide shade and weather protection. It has a sloping roof of corrugated asbestos with an opening the entire length of the building to allow any fumes to escape. The opening has a cover to prevent rain and the direct rays of the sun from entering. There shall be no expansion joints in the concrete. All construction joints shall be sealed with cement mortar. A loading platform and ramp are provided. A water-fog spray-piping system is installed around the periphery of the basin to control and dissipate any nitrogen tetroxide vapors. It is also used to flush away spills and for cooling the containers. A similar system is installed on the roof to control the fumes that will escape to the atmosphere through the ventilator in the roof.

7.11 Change House. (Dwg. F142-2-17)

This drawing shows details of a typical change house. It is large enough to provide the necessary facilities and floor space for six operator personnel. It is located inside the fenced-in area, but outside the "dyked" area near an entrance gate. The building is of permanent type construction of any of the following materials: brick, plaster, tile, corrugated sheet

asbestos, aluminum or steel with approved protective coating. The roof may be of any of the following materials: slate, corrugated steel or sheet asbestos, aluminum, or asbestos shingles. The conventional petroleum-based roofing materials are prohibited. Wooden or rubberized floors are prohibited. It is recommended that the floors be made of concrete. Local conditions may dictate a choice of the above-mentioned materials.

The building is divided into rooms providing space for an office, workshop, laboratory, and storage area. Two locker rooms, a shower room, and two toilets are included. Two sets of lockers will be required to provide each man with one locker for street clothes and one for work and safety clothing. Lockers for storing street clothing will be in a room separate from the lockers storing work and safety clothing. Spare protective equipment such as face masks, boots, gloves, suits, etc, will be kept in the storage room. Racks, cabinets, and cleaning facilities are included. Hot and cold water, heat, electricity, and telephone services must be provided. The office in the change house will be a permissible location for personnel to eat lunch. The change house will be the only permissible location for smoking inside the fenced area. Appropriate signs will be placed accordingly.

A master switch to control all of the electrical equipment in the facility should be located on the outside wall of the change house nearest the working area. This switch will be used to shut-off all electrical power in the working area, except the flood lights, the water pump motors, and the sump pump motor, in the event of an emergency. A separate master switch to control the above excepted items should be located inside the change house. An appropriate sign should be mounted above each switch indicating its function.

- 7.12 Liquid Disposal System (Nitrogen Tetroxide). (Dwg. E142-2-18)
- 7.12.1 General This section contains information on the design, construction, and operation of a liquid disposal system for use in conjunction with a nitrogen tetroxide bulk storage facility. The disposal system is used for holding and treating large quantities of off-specification or unwanted nitrogen tetroxide prior to discharging to a neutralizing system in the form of dilute nitric acid.

The system is based on the following theory: N_2O_4 is soluble in water, forming nitric and nitrous acids. The nitrous acid undergoes decomposition forming additional nitric acid and evolving nitric oxide (NO). However, the degree of solubility depends upon how completely it can be exposed to water. Theoretically, N_2O_4 reacts with water as follows:

$$N_2O_4 + H_2O = HNO_3 + HNO_2$$

The nitrous acid undergoes decomposition as follows:

$$3HNO_2 = HNO_3 + 2 NO + H_2O$$

As a result of the above two reactions, two-thirds of the N_2O_4 forms nitric acid; the other third combines with air to form nitrogen dioxide (NO_2). Therefore, when N_2O_4 is spilled into a pool or container of water a portion of it combines with water to form nitric acid; the remainder combines with air to form NO_2 .

7.12.2 Description of the System - (Dwg. E142-2-18) This drawing shows a detailed piping arrangement of the three 11,000-gallon tanks and the two centrifugal pumps used in the disposal system. The three tanks are manifolded together with a series of pipe headers and are connected to the two pumps in a manner designed for complete flexibility and interchangeability. Nitrogen gas is provided in the event transfer of liquid by the differential pressure method is desired. All the necessary valves and instrumentation are provided.

The system is connected to the storage and transfer system, the fume absorption system, and the neutralizing system.

7.12.3 Description of the Process - Basically, the process is simply the addition of nitrogen tetroxide to water, forming nitric acid and nitrogen dioxide. The nitric acid is then transferred to a neutralizing system and the nitrogen dioxide is piped to a fume absorption system.

In order to clarify the process, a typical disposal operation is described herein, but in general terms only. Detailed instructions are given in paragraph 7.13.1, Operating Instructions.

As an example, it is assumed that 10,000 gallons of nitrogen tetroxide has failed to meet the specification and must be disposed of. It is first transferred to the holding tank in the disposal system where it may be stored, prior to actual treating operations.

In order to begin these operations, it is first necessary to charge each of the two treating tanks with 5,000 gallons of water. (The liquid level gauge on each tank should show a reading of approximately 50% full.) The water is then recirculated by pumping from tank "A" to tank "B" and returning to tank "A" via the liquid balance line. This recirculation of water is continuous and must be maintained throughout the entire treating operation. The nitrogen tetroxide in the holding tank is also recirculated by pumping from the outlet header of that tank back through the inlet header via the meter by-pass header.

While both of the liquids are being recirculated, the nitrogen tetroxide is slowly fed into tank "A" through the flowmeter. At the completion of this operation, the contents of the treating tanks, which is now nitric acid, are transferred to the neutralizing system.

7.13 Liquid Disposal System (Nitrogen Tetroxide) Flow Diagram. (Dwg. F142-2-19)

This flow diagram identifies all manually operated valves by a number. All pipe lines and other equipment are identified by name. The flow diagram should be studied carefully, and all operators should have a thorough understanding of which valves must be used to perform a particular operation. Copies of this flow diagram should be posted at several conspicuous places. The reproductions (signs) shall be easily readable, durable, and they must be protected to prevent disfiguration or damage.

7.13.1 Operating Instructions.

7.13.1.1 General - Before any operation is started, make sure the disposal system is ready to operate and all the manually operated valves are closed.

During any transfer or treating operation, the fume absorption system shall

be operated as a falling film scrubber, (paragraph 7.14.5.4.1.b.). When the tanks are used for storage, the fume absorption system shall be operated as a static scrubber, (paragraph 7.14.5.4.1.a.).

To transfer nitrogen tetroxide to the holding tank:

- a. Use fume absorption system as a falling film scrubber.
- b. Open valve 1 and manual vent valve 4.
- c. Transfer nitrogen tetroxide to the holding tank.
- d. Close valves 1 and 4.
- e. Fume absorption system is now operated as static scrubber.

Although the holding tank has a capacity of 11,000 gallons, the maximum amount of nitrogen tetroxide that may be treated in one continuous operation is 5,000 gallons. However, it is not anticipated that this amount would have to be disposed of at any one time. The system is also capable of treating small quantities of nitrogen tetroxide.

7.13.1.2 <u>Treating Large Quantities</u> - The following are step-by-step instructions for a typical treating operation for 5,000 gallons of nitrogen tetroxide:

- a. Open manual vent valves 5 and 6.
- b. Open valves 9 and 10 in pressure balance line.
- c. Open valves 12 and 13 in the liquid balance line.
- d. Open water supply valve(s) 18 and/or 19. The two treating tanks are charged with water simultaneously until each tank is 50% full or approximately 5,000 gallons per tank.
- e. Close valve(s) 18 and/or 19.
- f. Open valves 21, 27, 35, and 41.
- g. Turn on pump No. 2 to circulate water between tank "A" and tank "B".
- h. Open valves 4 and 8 to relieve any pressure on the holding tank, and create a pressure balance between all three tanks.
- i. Open valves 23, 28, 32, and 36.
- j. Turn on pump No. 1 to circulate nitrogen tetroxide in the holding tank.
- k. Open valve 43.

1. Open valve 30 gradually to allow nitrogen tetroxide to pass through the flow meter into treating tank "A". The suggested flow rate is 5 gallons per minute. Valves 30 and 32 may have to be manipulated to get the desired flow rate.

NOTE: The suggested flow rate may be increased or decreased, depending upon operating conditions at the time. The rate of addition of nitrogen tetroxide to the water in the treating tanks is determined by three things:

- (1) The Pressure Build-Up in the Treating Tanks. The tank is designed to withstand an internal pressure of 150 psig. The rupture disc is rated at 75 psig. The automatic pressure valve is set to relieve at 70 psig. Therefore, a maximum working pressure of 60 psig is recommended. The pressure build-up is an indication of the amount of NO₂ generated.
- (2) The Volume and Rate the Nitrogen Dioxide (NO₂) is released to the Absorption System. When treating the NO₂ fumes from this operation, the fume absorption system is operated as a falling film scrubber. The operators must observe the density and color of the vapors expelled from the vent on the absorption column. These vapors should be wispy white. If the expelled vapors are yellow or brown, the absorption system is being overloaded, due to the excessive generation of NO₂ in the treating tanks. To correct this condition, the rate of flow of nitrogen tetroxide to the treating tanks must be reduced.

NOTE: If it is desired to determine the content of NO2 in the exhaust from the scrubber, a nitrogen dioxide detector and alarm system may be installed (paragraph 6.2).

(3) The Rate of Temperature Increase Within the Treating Tanks. The water temperature should be about 50°F at the start of the operation. It should not exceed 100°F at any time during the treating procedure. If the water gets too hot, the solubility of the nitrogen tetroxide in water is considerably decreased, releasing excess nitrogen dioxide to the scrubbing system. Excessive temperature increase is a direct result of feeding the nitrogen tetroxide too fast into the treating tank. If the reaction is too

great, the feed must be stopped. Experience will prove that a continuous gradual feed of nitrogen tetroxide is preferred. In the case of a violent reaction, it may be necessary to cool the mixture by diluting with additional water, and open valve 7 for a short period to relieve the excess pressure to the atmosphere.

- m. When the liquid level indicator on the N_2O_4 holding tank indicates 50% full or about 5,000 gallons delivered, close valves 43 and 30.
- n. Turn off pump No. 1. Close valves 4, 8, 23, 28, 32, and 36.
- o. Allow pump No. 2 to continue recirculating the liquid until the reaction between the N₂O₄ and the water has been completed. This is determined by the vapor pressure reading on the pressure gauges on the two treating tanks. As the reaction decreases, the pressure reading will be reduced proportionately until a constant reading is obtained. This constant reading cannot be predetermined but will be lower than that obtained during the treating period.
- p. Turn off pump No. 2. Close valves 21, 27, 35, and 41.
- q. The two treating tanks are now partially filled with dilute nitric acid.

NOTE: At this stage, the dilute nitric acid may be temporarily stored in the two treating tanks or it may be transferred in measured quantities to the neutralizing system.

If the liquid is to be stored in the treating tanks, valves 5, 6, 9, 10, 12, and 13 are left open. The fume absorption system is then operated as a static scrubber.

If the liquid is transferred to the neutralizing system proceed as follows:

- a. Close valves 5 and 6. Allow valves 9, 10, 12, and 13 to remain open.
- b. Open valves 21, 27, 31, 45, and 47.
- c. Put fume absorption system into operation as a falling film scrubber to handle the fumes from the neutralizing system.

- d. Turn on pump No. 2 and transfer a sufficient quantity of liquid (2,000 gallons maximum) to the receiving tank of the neutralizing system. The amount delivered will be indicated on the flow meter.
- e. When the receiving tank on the neutralizing system has been filled, turn off pump No. 2 and close valves 21, 27, 31, 45, and 47.

NOTE: At this point, the dilute nitric acid transferred to the receiving tank must be processed in the neutralizing system as described in paragraph 7.15. When this dilute nitric acid has been processed, repeat transfer operations outlined in instructions, a, b, c, d, and e above until the two treating tanks are empty.

If 10,000 gallons of nitrogen tetroxide has been originally transferred to the holding tank, the remaining 5,000 gallons must also be treated. Repeat the treating operations described above.

After the treating and disposal operations are complete, the system is shut down and secured by closing all manually operated valves and locking the two pump control switches.

- 7.13.1.3 Treating Small Quantities Small quantities of unwanted nitrogen tetroxide may be treated in a similar manner except that only one treating tank would be used. The mixing proportions are two parts of water to one part of nitrogen tetroxide.
- 7.13.1.4 Alternate Operating Procedures The disposal system is designed for maximum flexibility in the treating and transferring of liquid. The tanks and pumps are manifolded together by a series of headers providing the required interchangeability of tanks and pumps necessary for alternate operations or unusual situations due to equipment failure. It is possible, for instance, to use any one of the three tanks as a holding tank and the remaining two tanks for treating purposes. Likewise, the two pumps are also interchangeable, since either pump may be used for transferring or circulating. This flexibility of equipment is possible by using the various combinations of valves and headers provided.

Nitrogen gas is also provided and may be used to transfer liquid by the differential pressure method. For example, in order to transfer nitrogen tetroxide from the holding tank to treating tank "A" using nitrogen gas instead of a pump, open valves 14, 23, and 43. Then open valve 49 gradually, until the desired rate of flow of nitrogen tetroxide into the treating tank is obtained, as indicated on the flow meter. The same operating characteristics concerning pressure build-up, temperature increase, and NO2 fume release apply when using nitrogen gas as a transfer medium.

The <u>circulation</u> of liquid cannot be done by the differential pressure method. The nitrogen gas may also be used to fill the void created by removing nitrogen tetroxide from a tank.

Suction stubs are provided at each pump as a convenience for connecting portable equipment to the system. Valve 51 and 52 is used in this case, depending upon which pump is used.

A connection is also provided in the N₂O₄ inlet line for use in unloading nitrogen tetroxide from mobile tankers for disposal purposes only. Valve 53 is used for this purpose. A check valve is provided at this point to prevent any off-specification nitrogen tetroxide from flowing back to the storage tanks.

- 7.14 Fume Absorption System. (Dwg. E142-2-20)
- 7.14.1 General This section contains information on the design, construction, and operation of a fume absorption system for use in conjunction with a nitrogen tetroxide bulk storage facility. Neutralization and absorption of these fumes are necessary to prevent contamination of the atmosphere and subsequent danger to personnel.

The system has the capability of neutralizing a continuous high flow rate of fumes liberated during the transfer or treating of nitrogen tetroxide in tank car or tank truck lots and absorbing an intermittent low flow rate of fumes which may be emitted during normal storage. The absorption column is designed to accommodate fumes generated during the transfer of 11,000 gallons of nitrogen tetroxide at a maximum rate of 150 gallons per minute. For

normal storage conditions, the column will neutralize fumes generated from several 11,000-gallon tanks.

The information may be used as a guide to plan a nitrogen tetroxide (and nitric acid) fume absorption unit at any Air Force Base except in the Arctic Zone.

This information will be used as a guide only, since no attempt has been made to provide minute construction details. It should be stressed, however, that the principles of the piping system should be closely adhered to. It is recommended that pipe sizes as shown be used where indicated regardless of the size of the storage installation. This should be done so that all USAF installations may be standardized, thus simplifying the supply problem and providing for interchangeability of parts. The piping system contained herein provides for maximum flexibility during the operation of the fume absorption system. It is mandatory that the materials specified be used.

- 7.14.2 <u>General Criteria</u> On choosing a site for the erection of the fume absorption system, the following should be taken into consideration:
 - a. Available Water Supply Adequate water will be required for preparation of the scrubbing solution and for flushing and wash-down purposes. Each water outlet should have a flow rate of at least 50 gal. per minute for flushing or decontamination purposes.
 - b. Effluent Disposal A waste disposal system will be required to accommodate the disposal of spent scrubbing solution and wash-down water. This effluent can be accommodated through the regular sewer system.

7.14.3 General Requirements.

- a. Details of this system are shown on the following Chemical Corps drawings which form a part of this manual:
 - E142-2-20 Fume Absorption System
 - D142-2-21 Fume Absorption System Flow Diagram
 - E142-2-22 Absorption Column
 - E142-2-23 Scrubbing Solution Tank (Potassium Permanganate)
 - E142-2-26 Fume Absorption System Structural Details

- b. All piping used for transferring or circulating basic potassium permanganate scrubbing solution should be constructed from standard weight, Schedule 40, 18-8 stainless steel. Wherever possible, all connections throughout the piping system will be heli-arc welded. Wherever flanges are used, 18-8 stainless steel bolts will be used, utilizing an acceptable gasket material between flanges. Due to the unreliability of screwed connections, it is recommended that they not be used in major pipe circuits. Where screwed connections must be used, teflon tape thread sealant is recommended. A slurry of litharge and glycerine with 5% water may be used as a pipe dope. The litharge dope is "set" when its color changes from orange to white. This curing period is mandatory before using the system.
- c. Structural details showing the installation of the equipment used in the fume absorption system are shown on Dwg. E142-2-26.
- 7.14.4 <u>Laboratory Facilities</u> Space will be required for location of field laboratory facilities. It may be located in the change house or in a separate building. These facilities are required to aid in the preparation of scrubbing solution and for checking and controlling the process.

The basic laboratory equipment required will be as follows:

Laboratory balance

Platform scale - 0 to 1,000

Small portable electric mixer

Assorted beakers

Assorted graduated cylinders

Assorted pipettes

The basic chemicals required will be as follows:

Potassium permanganate - technical

Sodium hydroxide - technical

Sulfuric acid - CP

7.14.5 Operations.

- 7.14.5.1 Personnel Responsibilities The operating personnel of the fume absorption system shall be thoroughly indoctrinated in the hazards present when handling nitrogen tetroxide. It is mandatory that operating personnel be completely familiar with the operation of the fume absorption system, the method of checking the strength of the scrubbing solution and the procedure for preparing new solution. The operating personnel shall be thoroughly familiar with all the equipment and be able to perform minor repairs and maintain the equipment in good operating condition.
- 7.14.5.2 <u>Preparation of Scrubbing Solution</u> The scrubbing solution is prepared in the solution storage tanks in 350-gallon batches. It is also stored in these tanks until used. The following quantities are used in the preparation of 350 gallons of solution:

Potassium permanganate (KMnO₄) - - - - - 260 lb. Sodium hydroxide (NaOH) - - - - - - - 130 lb. Water (total) - - - - - - - 350 gal.

The scrubbing solution is prepared in accordance with Instructions a or b, paragraph 7.14.5.6.

The caustic (sodium hydroxide) must be handled carefully to prevent contact with the skin as severe burns will result. If caustic is spilled on the person, flood the affected area with water immediately.

7.14.5.3 Laboratory Test for Solution Strength - A simple and accurate test is described herein to determine if the scrubbing solution is spent, or should be renewed. Samples of scrubbing solution must be taken throughout the transfer period and the strength determined immediately. If the solution is spent, the reserve solution tank is placed in operation.

When a strongly basic solution of potassium permanganate (KMnO₄) is used as the scrubbing solution, it is necessary to determine when the concentration of potassium permanganate is too low and if the solution should be renewed. This test is simply the addition of dilute sulfuric acid (H₂SO₄) preferably 10N, until the solution is strongly acid, and noting if the permanganate color disappears. If the red permanganate color disappears, the solution should be changed. The amount of 10N sulfuric acid used need not exceed 10% of the sample tested, or 10 ml of dilute sulfuric acid to 100 ml of scrubbing solution. If the permanganate color disappears after several minutes, the scrubbing solution should be replaced by fresh solution. If the permanganate color remains, the solution is still usable (Ref. 7).

Since the manganese dioxide (MnO₂) precipitate will settle and become slightly packed upon long standing (in the scrubber), the solution should be circulated at least once a week to prevent sludging. If the scrubbing unit is to be inactive longer than several weeks, the solution should be drained out of the tanks and the entire system flushed with water.

7.14.5.4 Operation Procedure for Fume Absorption System.

7.14.5.4.1 General - The fume absorption column described herein may be used either as a "static" scrubber or as a "falling film" scrubber, depending upon operating conditions existing at the nitrogen tetroxide storage unit, the disposal system or other fume source.

The two conditions are as follows:

a. Static Scrubber - The column is used as a static scrubber under normal storage conditions, when there is a possibility of an "intermittent low flow rate" of fume discharge from the nitrogen storage tank(s). This would occur only when the internal pressure of the storage tank exceeded the pressure relief valve setting. The fumes would then be released to the scrubber until the internal tank pressure is relieved and the

- relief valve returns to its preset normally closed position. Under static conditions, the column contains a 4-ft level of scrubbing solution through which the escaping fumes must pass. The scrubber is always maintained in the static condition as a safeguard against pressure build-up in a storage tank when no transferring operations are taking place. See paragraph 7.14.5.6, Instruction c.
- Ъ. Falling Film Scrubber - The column is used as a falling film scrubber for fumes delivered at a continuous high volume rate such as those generated as a result of filling a tank with nitrogen tetroxide, or during treating operations in the disposal system. This high fume rate would occur if the displaced tank vapors were not returned to the source container, but were instead delivered to the scrubber. In this case, the manual vent valve on the N_2O_4 tank being filled is opened to allow all the exhausting NO_2 fumes to be piped to the scrubber. Under this condition, the scrubbing solution is circulated continuously by a pump from a storage tank to the top of the scrubber, down through the packed column, and back to the tank. The fumes must pass upward through this continuous downward flow of scrubbing solution. The fumes are released into the atmosphere through a vent on the column. At the end of the pumping operation, the column is filled to a 4-ft level of solution and is again used as a static scrubber. See paragraph 7.14.5.6, Instruction d.
- 7.14.5.4.2 Solution Tank Changeover During the Operation of the System as a Falling Film Scrubber During the course of operations, it is necessary to check the appearance of the scrubbing solution in the sight glass in order to determine if the solution is depleted. If the solution is a deep purple, it is an indication that it has retained its full strength and is satisfactory for use. However, if the solution is a pale shade of purple, a sample should be taken (valve 21 on Flow Diagram) and checked in the laboratory for strength (paragraph 7.14.5.3). If the solution is depleted in any one tank, that tank is cut out of the system and the other tank is cut in. This changeover is accomplished without interruption to the scrubbing operation (Instruction e, paragraph 7.14.5.6). The depleted solution is drained and a new batch of fresh solution is prepared.
- 7.14.5.4.3 Absorption Column Cleaning and Flushing Continued use of the absorption column will result in the gradual formation of MnO₂ deposits on the berl saddles. The saturated solution of potassium permanganate is

reduced by nitrogen dioxide, forming a heavy precipitate of manganese dioxide. If these deposits are not removed, the column will eventually become plugged.

In order to remove the deposits it is necessary to clean and flush the column. This is done by filling the column with water and bubbling nitrogen dioxide through the column of water, thereby acidifying the water. When the water is sufficiently acidified, the deposits will dissolve. The column is then drained, refilled with plain water, and again drained. It is then ready for a new charge of scrubbing solution.

If there are no nitrogen dioxide vapors generating in the storage tanks, it may be necessary to make up an acidified water solution using nitric acid. This may be prepared in the scrubbing solution make-up tank and pumped into the column. For detailed column cleaning instructions see paragraph 7.14.5.6, Instruction f.

7.14.5.5 <u>Miscellaneous Operating Techniques</u> - (Refer to Flow Diagram, Dwg. D142-2-21 for valve identification.)

- a. Always make sure that the scrubbing solution used is fresh and full strength. Never start a nitrogen tetroxide filling or transfer operation using depleted or low strength scrubbing solution.
- b. Dissolving the caustic (sodium hydroxide) in the water when making scrubbing solution will cause a temperature rise in the water. This condition is normal and desirable, since it accelerates the dissolving of the permanganate.
- c. The manual vent valves (9 and 18) on the solution tanks are normally closed except when filling the tanks with water or emptying a depleted charge.
- d. The effluent from the column or the tanks may be run to any effluent drainage system.
- e. The normal absorbent column pressure gauge reading is zero. A pressure build-up inside the column, as indicated on this gauge, indicates trouble and requires immediate investigation.
- f. In the event of a breakdown, pump A may be used to pump from tank B or vice versa. Pump by-pass valves 5 and 14 are provided for this purpose.

- g. Valve 21 is provided for sampling the scrubbing solution prior to laboratory inspection.
- h. A liquid trap and check valve are installed in the fume inlet line at the column to prevent the scrubbing solution from draining back to the fume source through the fume vent line.

7.14.5.6 Operating Instructions - Following are the basic operations involved:

- a. Solution Make-up Using Tank A.
- b. Solution Make-up Using Tank B.
- c. Operation of System as a Static Scrubber.
- d. Operation of System as a Falling Film Scrubber.
- e. Solution Changeover Procedures During the Operation of the System as a Falling Film Scrubber.
- f. Column Cleaning Procedure.

Step-by-step instructions for all the basic operations follow.

Refer to Flow Diagram, Dwg. D142-2-21 for identification of valve numbers and equipment.

- a. Solution Make-up Using Tank A.
 - (1) All valves closed.
 - (2) Open valve 9.
 - (3) Open valve 22. Fill with water to 350-gallon level indicated on sight glass.
 - (4) Close valves 22 and 9.
 - (5) Open hatch cover.
 - (6) Charge tank with 130 pounds of sodium hydroxide.
 - (7) Open valves 1, 3, 4, and 7.
 - (8) Start pump and circulate for 20 minutes.
 - (9) Charge tank with 260 pounds of potassium permanganate.
 Allow pump to circulate this solution for one hour.
 - (10) Turn off pump and close all valves and hatch cover.
 - (11) The solution in Tank A is now ready for use in the scrubbing tower (absorption column).

b. Solution Make-up Using Tank B.

- (1) All valves closed.
- (2) Open valve 18.

- (3) Open valve 23. Fill with water to 350-gallon level indicated on sight glass.
- (4) Close valves 23 and 18.
- (5) Open hatch cover.
- (6) Charge tank with 130 pounds of sodium hydroxide.
- (7) Open valves 10, 12, 13, and 16.
- (8) Start pump and circulate for 20 minutes.
- (9) Charge tank with 260 pounds of potassium permanganate.
 Allow pump to circulate this solution for one hour.
- (10) Turn off pump and close all valves and hatch cover.
- (11) The solution in Tank B is now ready for use in the scrubbing tower (absorption column).

c. Operation of System as a Static Scrubber.

- (1) All valves closed.
- (2) Using Tank A, open valves, 1, 3, and 6.
- (3) Start pump.
- (4) Allow the pump to remain in operation until the solution has reached the 4-ft level in the column as indicated on the upper liquid level indicator.
- (5) Stop the pump.
- (6) Close all valves.
- (7) The column may now be used as a static scrubber.

NOTE: When using Tank B, substitute valves 10, 12, and 15 for valves 1, 3, and 6.

d. Operation of System as a Falling Film Scrubber - (Using Tank A).

- (1) All valves closed.
- (2) Open valves 1, 3, 6, 19 and 8.
- (3) Adjust valve 6 so that the flow rate of the solution through the column is sufficient to maintain a liquid seal below the fume inlet in the column as shown on the lower liquid level indicator. If the flow rate of the pump is not sufficient to maintain a liquid seal in the bottom of the column with valve 19 fully open, then valve 19 must be throttled until this liquid seal is maintained. Readings may be taken on the flow meter for future use in pre-setting the flow.

- (4) The column may now be used as a falling film scrubber.
- (5) At the completion of the nitrogen tetroxide transfer operation, continue running the system for 30 minutes to insure absorption of all fumes generated.
- (6) At the end of this 30-minute period, close valve 19.
- (7) Allow the pump to remain in operation until the solution has reached the 4-ft level in the column as indicated on the upper liquid level indicator.
- (8) Stop the pump.
- (9) Close all valves.
- (10) The column may now be used as a static scrubber.

NOTE: When using tank B, substitute valves 10, 12, 15, and 17 for valves 1, 3, 6, and 8. Valve 19 is used in the same manner for both tanks.

e. Solution Changeover Procedures during the Operation of the System as a Falling Film Scrubber.

- (1) Assume tank A is in use but solution is depleted.

 Tank B is filled with fresh solution and is ready for use.
- (2) Tank A in use means that valves 1, 3, 6, 19, and 8 are open and pump A is running.
- (3) Open valves 10, 12, 13, and 16 at tank B.
- (4) Start pump B to circulate the solution.
- (5) Open valves 4 and 7.
- (6) Close valve 6 and open valve 15.
- (7) Open valve 17.
- (8) Close valve 8 on tank A.
- (9) Close valves 13 and 16.
- (10) Turn off pump A.
- (11) Close valves 1, 3, 6, 4, and 7.
- (12) Adjust valves 15 and/or 19 for proper flow rate described in Instruction d.
- (13) Open valves 1, 2, and 9 to drain tank A. When tank is drained, close valves 1, 2, and 9. Prepare fresh scrubbing solution in tank A (see Instruction a).

f. Column Cleaning Procedure.

- (1) All valves closed and assume tank A is empty.
- (2) Open valve 22 and fill to sight glass level.
- (3) Close valve 22.
- (4) Open valves 1, 3, and 6.
- (5) Start pump A, pumping the water into the column.
 Allow the pump to run until the water overflows from the vent pipe at the top of the column, then stop pump.
- (6) If there is a vapor pressure build-up in a nitrogen tetroxide storage tank, open the storage tank manual vent line and allow the nitrogen dioxide fumes to bubble through the column. When the water is sufficiently acidified, the deposits in the column will dissolve.
- (7) This condition is evidenced by the emission of yellowishbrown fumes from the column vent. When these fumes are observed, turn off the manual vent valve on the nitrogen tetroxide storage tank.
- (8) Open valve 20 and drain off acidified water.
- (9) Close valve 20.
- (10) Start pump A, pumping the water into the column.

 Allow the pump to run until the water overflows from the vent pipe at the tope of the column, then stop pump.
- (11) Open valve 20, draining off the water in the column.
- (12) Close valve 20.
- (13) Close valves 1, 3, and 6. All valves are now closed. Water remaining in the solution tank may be used for preparing the next batch of scrubbing solution.

NOTE: Tank B and pump B may also be used by substituting valves 23, 10, 12, and 15 for valves 22, 1, 3, and 6.

7.14.6 Specifications for Specific Materials.

7.14.6.1 Column Absorption.

a. General Requirements - This specification covers a NO₂
absorption column capable of neutralizing the fumes
liberated during the transfer or filling of an 11,000-gallon
storage tank at a maximum rate of 150 gpm of nitrogen

tetroxide. The column shall be constructed from stainless steel. This column is shown on CmlC Dwg. E142-2-22 which forms a part of this specification.

The column shall be designed, constructed, and tested in accordance with the ASME Code.

b. Materials and Workmanship - The equipment shall be new construction; material and workmanship shall be of the best quality, free from any defect that would render it unsuitable or inefficient for the purpose for which it is to be used. Any fault due to design, material or workmanship which may develop prior to the completion of the first year of service shall be adjusted by and at the expense of the contractor.

All welding used in the construction of this equipment shall conform to the best practices recommended by the American Welding Society.

c. Detail Requirements.

- (!) Arrangement The column shall be furnished complete with all parts as shown on CmlC Dwg. E142-2-22 which forms a part of this specification.
- (2) Design The column shall be designed for the following maximum allowable working pressures and maximum and minimum temperature conditions:

Pressure: Shell, internal - 0 to 25 psig

Temperature: Shell, maximum - 160°F
minimum - -65°F

The allowance for corrosion, erosion, and abrasion as required by the ASME Code shall be as follows:

Shell and Heads - 1/16-inch.

The column supports shall be designed for a maximum weight calculated by adding 5000 lb to the actual weight of the empty column.

- (3) Materials The material used in the fabrication of this column shall be at least equal in quality to 18-8 stainless steel type 316.
- (4) Fabrication The column shall be fabricated by inert gas shielded arc welding throughout. The finished welds shall be free from any foreign material. All flanged surfaces forming a part of this item shall be level and true after welding. Holes for bolts shall straddle their natural centerlines.

- Nameplate The manufacturer's name, maximum allowable working pressure, maximum allowable temperature, manufacturer's serial number, year built, and materials of construction shall be permanently affixed to the column by nameplate or other means which will resist damage. Other individual units of this equipment shall be furnished with the manufacturer's name, ratings, model, style, serial number, and all other data permanently affixed by nameplate or other means which will resist damage.
- (6) Intent All work and materials shown or noted on the drawings shall be executed and supplied by the contractor the same as if fully described herein. The contractor shall not take advantage of any omission of details required for completion of this item within the full intent of this specification.
- d. Inspection and Test This pressure vessel shall be tested by the manufacturer in accordance with the ASME Code and the contractor shall certify that the vessel has successfully passed all tests. All longitudinal and circumferential welded joints shall be radiographically examined as provided for in the ASME Code. The item shall be inspected, upon arrival at its destination, by an authorized Government Inspector, for compliance with this specification before final acceptance.
- e. Finish and Protection The interior surfaces of the tank shall be free from scratches and other surface imperfections. Excess weld metal shall be removed. The finished column shall be cleaned thoroughly to insure the removal of oil, grease, grinding, or machining compound, or other foreign material. All parts of the column shall be adequately protected to prevent corrosive attack or physical damage during shipment. All external parts shall be protected in accordance with the best commercial practices for equipment of this kind. All openings shall be enclosed by blank flanges and adequately protected against dirt, water, chemical or mechanical injury.

f. General Data.

ASME	para U69
Maximum Working Pressure Shell, Internal	25 psig
Maximum Operating Temperature	160°F
Minimum Operating Temperature	-65 ⁰ F
Corrosion Allowance	1/16 in.
Estimated Weight: Empty Maximum on Supports	2500 lb 4300 lb 14 ft
Size: Overall Height Outside Diameter	2 ft

7.14.6.2 Tank, Scrubbing Solution (Potassium Permanganate).

a. General Requirements - This specification covers a chemical solution storage tank of approximately 375 gal. capacity. The tank shall be fabricated from stainless steel. The tank is intended for vertical, above-ground installation. This tank is shown on CmlC Dwg. E142-2-23 which forms a part of this specification.

The tank shall be designed, constructed, and tested in accordance with the ASME Code.

b. Materials and Workmanship - The equipment shall be new. Construction, material, and workmanship shall be of the best quality, free from any defect that would render it unsuitable or inefficient for the purpose for which it is to be used. Any fault due to design, material, or workmanship which may develop prior to the completion of the first year of service shall be adjusted by and at the expense of the contractor.

All welding used in the construction of this equipment shall conform to the best practices recommended by the American Welding Society.

c. Detail Requirements.

- (1) Arrangement The tank shall be furnished complete with all parts as shown on CmlC Dwg. E142-2-23 which forms a part of this specification.
- (2) Design The tank shall be designed for the following maximum allowable working pressures and maximum and minimum temperature conditions:

Pressure: Shell, Internal - 0 to 45 psig

Temperature: Shell, Maximum - 160°F

Minimum - -65°F

The allowance for corrosion, erosion and abrasion as required by the ASME Code shall be as follows:

Shell and Heads - 1/16-inch

The tank supports shall be designed for a maximum weight calculated by adding 5000 lb to the actual weight of the empty tank.

(3) Materials - The material used in the fabrication of this tank shall be at least equal in quality to 18-8 stainless steel type 316.

- Fabrication The tank shall be fabricated by inert gas shielded arc welding throughout. The finished welds shall be free from any foreign material. All flanged surfaces forming a part of this item shall be level and true after welding. Holes for bolts shall straddle their natural centerlines.
- Nameplate The manufacturer's name, maximum allowable working pressure, maximum allowable temperature, manufacturer's serial number, year built, and materials of construction shall be permanently affixed to the tank by nameplate or other means which will resist damage. Other individual units of this equipment shall be furnished with the manufacturer's name, ratings, model, style, serial number, and all other data permanently affixed by nameplate or other means which will resist damage.
- (6) Intent All work and materials shown or noted on the drawings shall be executed and supplied by the contractor the same as if fully described herein. The contractor shall not take advantage of any omission of details required for completion of this item within the full intent of this specification.
- d. Inspection and Test This pressure vessel shall be tested by the manufacturer in accordance with the ASME Code and the contractor shall certify that the vessel has successfully passed all tests. All longitudinal and circumferential welded joints shall be radiographically examined as provided for in the ASME Code. The items shall be inspected, upon arrival at its destination, by an authorized Government Inspector, for compliance with this specification before final acceptance.
- e. Finish and Protection The interior surfaces of the tank shall be free from scratches and other surface imperfections. Excess weld metal shall be removed. The finished tank shall be cleaned thoroughly to insure the removal of oil, grease, grinding or machining compound, or other foreign material. All parts of the tank shall be adequately protected to prevent corrosive attack or physical damage during shipment. All external parts shall be protected in accordance with the best commercial practices for equipment of this kind. All openings shall be enclosed by blank flanges and adequately protected against dirt, water, chemical, or mechanical injury.

f. General Data.

ASME Code		paragraph U69
Maximum Working Shell, Internal	Pressure	45 psig
Maximum Operatin	g Temperature	160°F
Minimum Operating	g Temperature	-65 ⁰ F
Corrosion Allowand	e	1/16 inch
Approximate Capac	ity	375 gal.
Estimated Weight:	Empty	1000 1ь
	Maximum on Supports	44 00 lb
Size: Overall Heigh	ht	76 inch
Outside Diam	neter	48 inch

7.14.6.3 Berl Saddles.

a. Specification - Saddles, Berl for use in NO₂ Absorption Column shall be of acid-resistant material. Size: 1 inch.

b. Possible Source.

Maurice A. Knight, Akron, Ohio U.S. Stoneware Company, Akron, Ohio

7.14.6.4 Gaskets.

a. Specification - Gaskets, Teflon, plain or envelope type for 1,2, and 3-inch, 150-lb ASA raised-face flanges.

Sizes: 1 inch, 2 inch, and 3 inch.

b. Possible Source.

Melrath Supply and Gasket Co., Inc. Philadelphia 34, Pa.

United States Gasket Company Camden, New Jersey

Chemical and Power Products Company New York 4, New York

7.14.6.5 Flowmeter.

a. Specification - Flowmeter, for determining flow rate of basic potassium permanganate scrubbing solution, shall be of the flowrator type of stainless steel construction. A stainless

steel float and teflon packing shall be used. Range 0.0 to 50 gpm at a specific gravity of 1.0. Connecting outlets shall be 1-inch, 150-lb ASA raised-face flanges.

b. Possible Source.

Fisher and Porter Company, Hatboro, Pa.
Foxboro Instrument Company, Foxboro, Mass.

7.14.6.6 Circulating Pump.

a. Specification - Pump, centrifugal, stainless steel construction, for basic potassium permanganate scrubbing solution service, 2-in. suction, 1-1/2-in. discharge, adaptors for 1-in. flanged connections, open impeller, capable of handling 50 gpm to a TDH of 30 ft when running at 1150 rpm, complete with base. Pump will be directly connected to a 3-phase, 60-cycle, 220/440-volt, 1150-rpm standard totally enclosed motor with HP for entire range of pump. All exterior metal of pump, motor, and base to be painted with acid-resistant paint. Pump shall have standard chemical mechanical seals. (Example - John Crane Number 9)

b. Possible Source.

The Durion Company, Inc., Dayton 1, Ohio Aurora Pump Company, Aurora, Illinois Lawrence Pumps, Inc., Lawrence, Mass. Worthington Corp., Harrison, New Jersey

7.14.6.7 Pressure Gauge.

a. Specification - Gauge, pressure, indicating type, for use with NO₂ vaprs, 4-1/2-in. dial, cast iron body with acid-proof finish, 0-25 psi range, etched stainless steel 270° concentric dial, beryllium copper pressure spring, bottom male 1/4-in. forged steel tapered pipe thread with wrench flats and stainless steel Bourdon tube.

b. Possible Source.

Foxboro Instrument Company, Foxboro, Mass. Taylor Instrument Company, Rochester, New York Weston Instrument Company, Newark, New Jersey Manning, Maxwell and Moore, Bridgeport, Conn. Durion Pump Company, Dayton 1, Ohio

7.14.6.8 Valves, Gate.

a. Specification - Valve, gate, 150-lb, 1, 2 and 3-in. to handle basic potassium permanganate solution, shall be of type 316 stainless steel. Valve shall have 150-lb ASA raised-face flanges or threaded connections as required.

b. Possible Source.

Crane Company, Chicago, Illinois
William Powell Company, Cincinnati 22, Ohio
Cooper Alloy Corporation, Hillside, New Jersey
Alloy Steel Products Company, Inc., Linden, New Jersey

7.14.6.9 Valves, Globe.

- a. Specification Valve, globe, 150-lb, 1/4 in. to 1 in. to handle basic potassium permanganate solution shall be of type 316 stainless steel, valve shall have threaded connections.
- b. Possible Source.

Crane Company, Chicago, Illinois
William Powell Company, Cincinnati 22, Ohio
Cooper Alloy Corporation, Hillside, New Jersey
Alloy Steel Products Company, Inc., Linden, New Jersey

7.14.6.10 Fittings, Flanged.

- a. Specification Fittings, flanged, type 316 stainless steel, 1, 2 and 3-in. standard weight, Schedule 40, 150-lb ASA raised-faced, standard iron pipe sizes. Pipe flanges shall be 150-lb ASA raised-faced welding neck type.
- b. Possible Source.

Crane Company, Chicago, Illinois
Tube Turns, Louisville 1, Ky.
Cooper Alloy Corporation, Hillside, New Jersey
Watson-Stillman Fittings Division, H. K. Porter Company,
Inc., Roselle, New Jersey

7.14.6.11 Fittings, Threaded.

a. Specification - Fittings, threaded, 1-in. type 316 stainless steel, standard weight, Schedule 40, with standard tapered pipe thread.

b. Possible Source.

Crane Company, Chicago, Illinois
Watson-Stillman Fittings Division,
H. K. Porter Company, Inc., Roselle, New Jersey
Cooper Alloy Corporation, Hillside, New Jersey

7.14.6.12 Liquid Level Gauge.

- a. Specification Gauge, liquid level, with guard and glass tube, 150-lb, type 316 stainless steel, 1/2-in. size with 14-in. centers, 1/2-in. tapered pipe thread connections.
- b. Possible Source.

Crane Company, Chicago, Illinois
Jerguson Gage and Valve Company, Somerville, Mass.

7.14.6.13 Piping.

Specification - Pipe, 1/4, 1, 2 and 3-inch, type 316 stainless steel, schedule 40, standard weight, standard iron pipe sizes, 20 -ft cut lengths. The length of each piece shall not be less than that specified and shall not exceed the specified length by more than 1/4 inch. Pipe shall be supplied in the mill finish and shall be of uniform quality and temper, sound and free from injurious defects. It shall be reasonably straight. Pipe will have plain ends, suitable for field welding or for threading. Ends of pipe will be suitably protected from damage in transit. Inspection of the pipe may be made at the manufacturers plant or at the point where the material is received at the option of the Government. If the inspection is made at the manufacturers works, the inspector representing the Government shall be afforded all reasonable facilities without charge, to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Material failing to conform to these specifications or in which defects are discovered during manufacturing operations may be rejected. If material is rejected the manufacturers liability shall be limited to replacing the rejected material without charge to the Government. All rejected material will be returned to the manufacturer.

b. Possible Source.

Crane Company, Chicago, Illinois
Tube Turns, Louisville, Ky.
Babcock and Wilcox Company, New York, N. Y.

- 7.15 Acid Neutralizing System (Dwg. E142-2-24).
- 7.15.1 General This section contains information on the design, construction, and operation of an acid neutralizing system used in conjunction with a nitrogen tetroxide bulk storage facility. The system is used to neutralize nitric acid prior to discharging to a sewer.
- 7.15.2 Description of the System (Dwg. E142-2-24) This drawing shows a detailed piping arrangement of a 2000-gallon receiving tank, a neutralizing bed, and a sump well. This equipment is connected to two pumps with all the necessary valves and instruments. The neutralizing bed consists of several sections of various sized stainless steel sections containing limestone or dolomite. The bed is constructed to provide a cascading method of slowly neutralizing the acid. A pH detection and control system and motor controller is installed in the sump well. It is used to measure the acid content of the liquid after the acid has passed through the neutralizing bed, and to turn on the recirculating pump. A positive displacement type pump is used for recirculating. A sump pump is provided to drain the sump well.
- 7.15.3 Description of the Process Nitric acid is pumped from the disposal system to the receiving tank from which it is allowed to flow by gravity through the neutralizing bed. After passing through the neutralizing bed the liquid is collected in the sump well. The pH detector, installed in the sump well, measures the acid content of this liquid. If the acid content is low, (pH reading above 5) the liquid is disposed of by pumping it to a sewer. If the acid content is high, (pH reading below 5) the liquid is automatically pumped back to the receiving tank from which it is allowed to flow through the neutralizing bed a second time.
- 7.15.4 Operating Instructions Before any operation is started the operator shall make sure the neutralizing bed is charged with the proper amount of limestone or dolomite. Each section must be filled to a depth of approximately two feet. The operator shall also make sure all manually operated valves are closed and the automatic pressure relief valve is set to relieve at 70 psig. During any transfer or neutralizing operation the fume absorption system shall be operated as a falling film scrubber. Since the

receiving tank has a capacity of only 2000 gallons, this is the maximum amount of nitric acid that may be transferred at any one time. The amount of nitric acid in the disposal system will determine the number of batches that must be transferred.

To transfer nitric acid to the receiving tank, using a pump in the disposal system, proceed as follows:

- a. Use fume absorption system as a falling film scrubber (paragraph 7.14.5.4.1.b.).
- b. Open valve 1 and manual vent valve 2.
- c. Transfer 2000 gallons of nitric acid to the receiving tank.
- d. Close valves 1 and 2.

To transfer nitric acid to the receiving tank, using the pump in the neutralizing system:

a. Proceed as outlined in 1, 2, 3, and 4 above, except use valve 6 instead of valve 1.

To neutralize the acid proceed as follows:

- a. Open valve 3 to allow air to enter the tank.
- b. Open valve 5 to allow the acid to flow into the neutralizing bed.
- c. Open valves 2 and 7 to allow the acid to be recirculated in the event pH detector turns on the pump.
- Cleaning and Charging the System Whenever the limestone or dolomite in a section of the neutralizing bed becomes spent, the system must be shut down and the section or sections thoroughly cleaned. The operator must first fill the bed with water, using a hose connected to valve 14. Then the drain valve is opened and the section is thoroughly flushed. Only the drain valves in the sections to be cleaned are opened. When a section has been thoroughly flushed with water the iron carbonate residue remaining in the section must be removed with a shovel. This residue may be disposed of at any convenient place. The section must be recharged with fresh limestone or dolomite to a depth of two feet.

Sodium carbonate may be added to the limestone or dolomite to help the neutralizing process. Any water contained in the sump well must be pumped to a sewer. Valve No. 4 on the receiving tank is used to drain the tank.

During the neutralizing procedure the operator should occasionally make a test of each section of the neutralizing bed to determine the acid content of the liquid. The operator must open the "thief hatch," provided in the cover of each section of the neutralizing bed, and submerge a piece of pH paper. If this test shows the liquid in any of the sections to be below the allowable safety limit, the operator may cut out the remaining sections of the neutralizing bed by opening any of the drain valves numbered 8 through 13. This will allow the liquid to by-pass any section(s) of the bed and flow directly to the sump well.

When all the acid in the receiving tank has passed through the neutralizing bed and is collected in the sump well, the operator shall close all valves. The operator will then turn on the sump pump to discharge all the liquid in the sump well to a sewer. When all the liquid is pumped out of the sump well the neutralizing system is ready to be used again. If there is no more acid to be neutralized, the operator must open the drain valve of each section used so that any trapped liquid will drain to the sump well. However, before the system is used again the operator shall inspect all sections of the neutralizing bed. If the limestone or dolomite in any of the sections is spent, the operator shall recharge that section with fresh limestone or dolomite as outlined in paragraph 7.15.5.

7.16 Sump Pump Installation (Dwg. F142-2-25).

This drawing shows a detailed arrangement of a sump well. It is used for collecting liquids from the operating stations and drain basis in the facility and transferring the liquid either to a sewer or to the disposal system. The liquids may be rain water, cooling water, nitrogen tetroxide, or nitric acid formed as a result of flushing nitrogen tetroxide spills with water. The sump well is connected to the several drainage basins by underground piping.

The sump well consists of a 1000-gallon stainless steel tank installed in the ground as shown on Dwg. F142-2-25. The tank has a flanged top and two side outlet connections. One connection is the inlet and the other one is an overflow. A sump pump is mounted on top of the tank and is automatically operated by a float switch. The sump pump has two manually operated valves located in the discharge pipe. One valve allows the liquid to be pumped to a sewer and the other valve allows the liquid to be pumped to the disposal system.

During any operation involving the transfer of nitrogen tetroxide, at any operating station of the facility, the valve in the pipe line to the disposal system shall be opened to allow any contaminated liquid collected in the sump well to be pumped to the disposal system. The valve in the pipe line to the sewer shall then be closed. When the facility is not in operation, such as overnight, the valve in the pipe line to the sewer shall be opened to allow any uncontaminated water collected in the sump well to be pumped to the sewer. The valve in the pipe line to the disposal system shall then be closed.

Whenever an emergency occurs at any operating station of the facility requiring the operation of the water-fog spray system, the operator shall make sure the sump well is put into operation so that all liquid collected in the sump well will be pumped to the disposal system.

8. SHIPPING.

8.1 Applicable Laws.

This section is intended only as a guide for the user. Extracts from the code are condensed and rephrased in the interest of brevity. For complete and official information and specifications, the user is referred to the Code of Federal Regulations, Title 49, Parts 71 to 90 and the latest supplements thereto. This code is available from the Superintendent of Documents, Washington 25, D. C.

8.2 Classification and Label.

Nitrogen tetroxide is classified by ICC as a Class A poison, no exemptions, and containers require a red label (Chemical NOIBN).

8.3 Packaging.

Nitrogen tetroxide is shipped in high pressure seamless steel cylinders and single unit tank cars to the following specifications:

ICC - 3D 480 - 10 lb net and 156 lb net

ICC - 3A 2015 - 13 lb net

ICC - 3A 1800 - 125 lb net

ICC - 106A 500-x 2000 lb net

Tank Cars.

ICC - 105A 500-W-10,000 gal

8.4 Procedure in the Event of Shipping Accident.

Normal safety precautions usually taken in the handling of any toxic or potentially poisonous material should be strictly observed, with special emphasis being given to avoiding the inhalation of the toxic fumes and recognition of the fact that nitrogen tetroxide will not burn but will vigorously support combustion. In case of spillage, personnel should immediately be evacuated from the area and the entire area washed down with copious quantities of water.

9. RECOMMENDED SAFETY INSTRUCTIONS.

The following safety instructions should be prominently displayed at numerous locations at all nitrogen tetroxide storage and handling installations.

SAFETY INSTRUCTIONS FOR NITROGEN TETROXIDE

HAZARDS

- 1. Skin contact causes severe burns.
- 2. Breathing of vapor may cause poisoning.
- 3. Spills may cause fire and/or liberate toxic gas.
- 4. Contact with fuels may cause explosions.

FIRST AID

- 1. Remove casualties from contaminated area. Apply artificial respiration if breathing has stopped, preferably with the aid of oxygen. Call for medical aid.
- 2. If nitrogen tetroxide gets into the eyes, flush them immediately with water, and continue flushing them for 15 minutes, holding the eyes open. If it is necessary to choose between treating the eyes and summoning medical assistance, wash the eyes for 10 minutes, call for medical attention, then resume eye flushing. Do not put anything but water in the eyes.
- If nitrogen tetroxide has been swallowed, drink large amounts of water, or milk if it is readily available.
- 4. If nitrogen tetroxide gets on the skin, remove any contaminated clothing and wash affected areas with large amounts of water.

9.1 Safety Instructions.

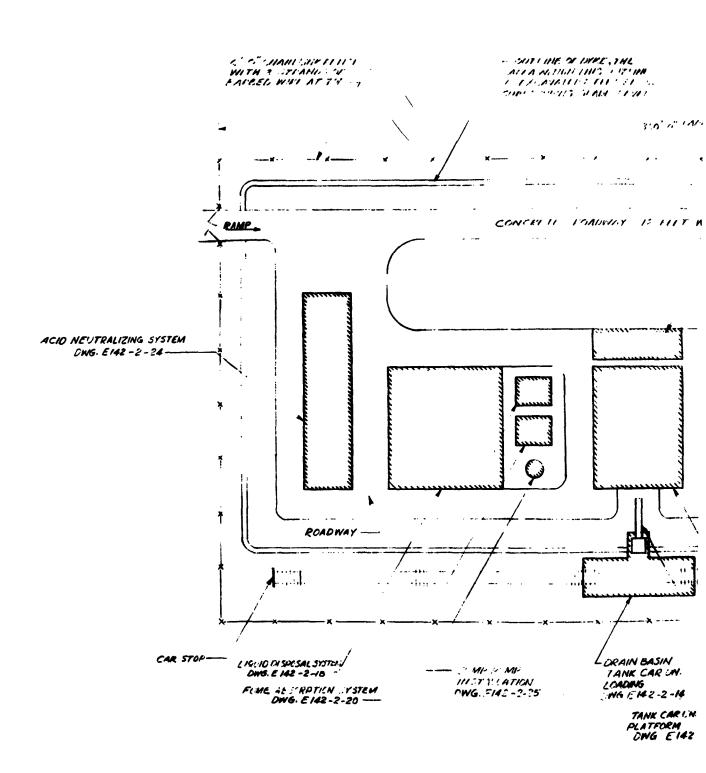
- a. The nature and characteristics of nitrogen tetroxide shall be explained to all persons working with this material.
- b. Persons engaged in operations involving handling or transfer of nitrogen tetroxide shall wear the approved boots, gloves, hood, and protective suit. In addition, a protective mask shall be worn by all persons exposed to nitrogen tetroxide resulting from spills. Persons entering confined spaces, where the atmosphere is contaminated with vapors of nitrogen tetroxide shall wear the approved self-contained breathing apparatus.

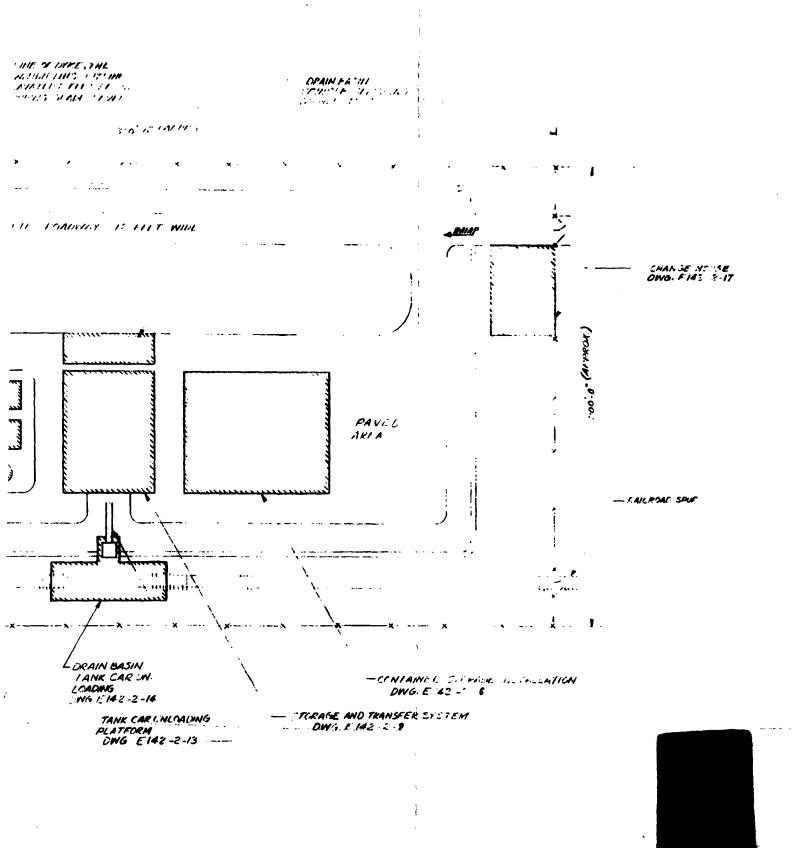
- c. Operations requiring the handling or use of nitrogen tetroxide shall be performed by persons working in groups of two or more.
- d. Before beginning any work on equipment, make sure the system is not pressurized. Work from above or on one side rather than from below a nitrogen tetroxide line. Avoid trapping nitrogen tetroxide between closed valves. Do not operate pumps against closed valves. Check lines, valves, and receiving tank before starting on nitrogen tetroxide transfer. Do not start repairs without permission of supervisor. Lock the switches to pumps and electrical lines under repair. Do not make nitrogen tetroxide transfers through lines being repaired.
- e. Safety showers, eye wash fountains, and personal protective equipment shall be inspected periodically, and prior to any operation involving nitrogen tetroxide.
- f. Use care when opening cylinders. Cylinders not manifolded must not be opened unless contents are below the boiling point (70°F at one atmosphere pressure).
- g. Avoid Spills Nitrogen tetroxide in contact with organic materials such as sawdust, excelsior, wood scraps, cotton waste, etc, may cause fire. Toxic fumes are generated from such spills, and color is not a reliable indication of toxicity.
- h. Protective clothing, hand tools, and other equipment, shall be flushed with water immediately after contact with nitrogen tetroxide.

10. REFERENCES.

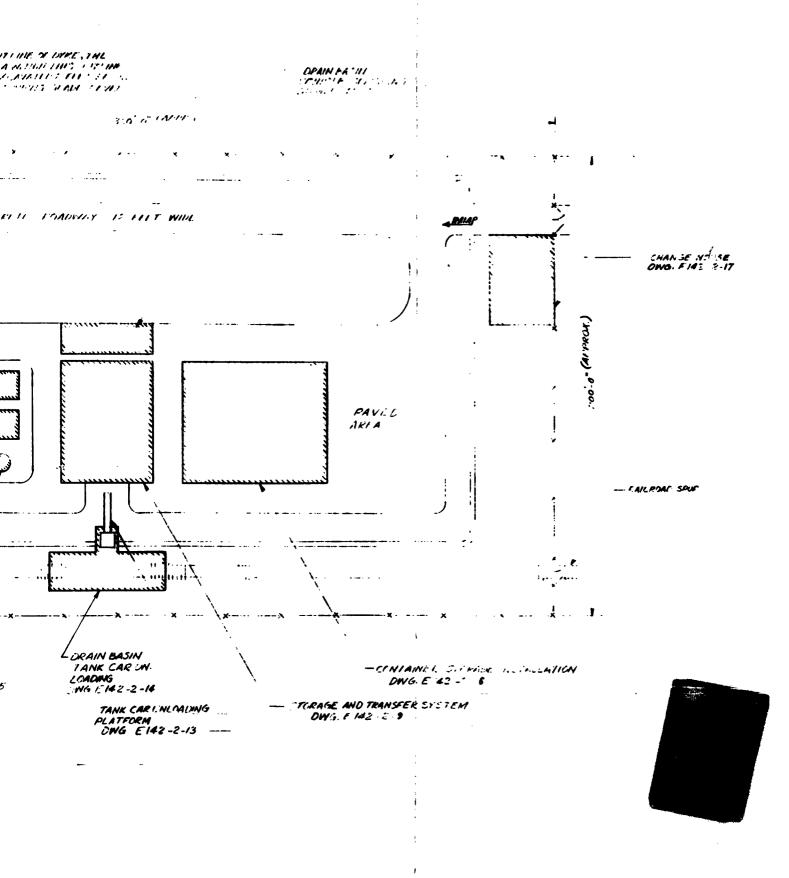
- 1. Handling and Storage of Liquid Propellants, Liquid Propellant Information Agency, March 1961.
- 2. Henderson, P., Storage and Handling of Red and White Fuming Nitric Acid, CRLR 372, U.S. Army Cml Res and Dev Labs, Army Cml Center, Md., 20 August 1954.
- 3. Ordnance Corps, Dept. of the Army, Ordnance Safety Manual, 20 February 1959.
- 4. Nitrogen Division of Allied Chemical, Nitrogen Tetroxide, June 1958.
- 5. Liberto, R. R., Storable Propellant Data for the Titan II Program, October 1960.
- 6. Health Hazards from Propellant Fuels and Oxidizers.
 Departments of the Army, Navy and Air Force. TB MED
 242, Nav Med P-5035, AFP 160-6-3.

- 7. Fleming, J. and Marine, G., An Evaluation of Methods and Equipment for the Absorption and Neutralization of Nitrogen Dioxide by Sodium Carbonate, Sodium Dichromate and Potassium Permanganate. CRLR 439, U.S. Army Cml Res and Dev Labs, Army Cml Center, Md., 7 January 1955.
- 8. Air Installation Division, Technical Services Section, Liquid Fuels Facilities Unit, T. O. 37B1-1-101, Handbook of Operation Maintenance and Inspection Instructions for Bulk Furning Nitric Acid Systems, Permanently Installed.
- 9. Henderson, P., A Test Series Conducted to Determine the Feasibility of Using Water Fog or a Strong Thermal Updraft to Control or Disperse the Fumes Resulting from Spills of Nitrogen Tetroxide. Tech Memo 31-113, U.S. Army Cml Res and Dev Labs, Army Cml Center, Md., 10 August 1961.

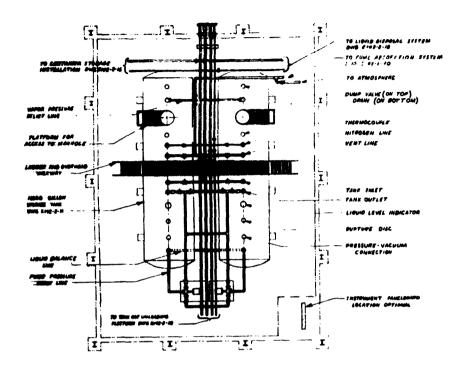


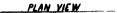


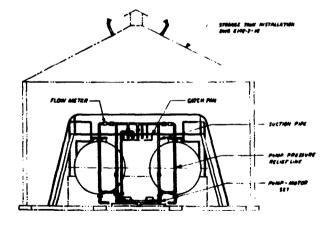
F142-2-8 Bulk Storage Facility - Plot Plan



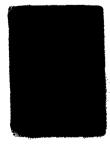
F142-2-8 Bulk Storage Facility - Plot Plan

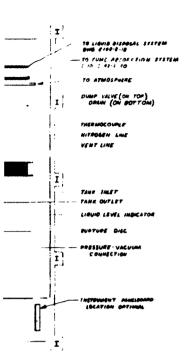


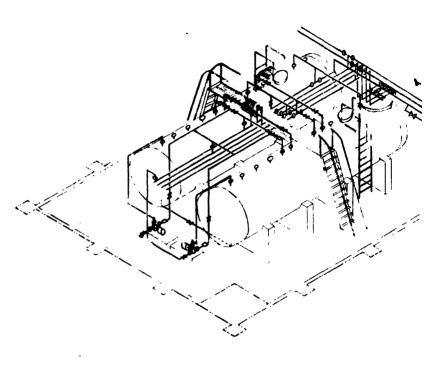




FRONT ELEVATION

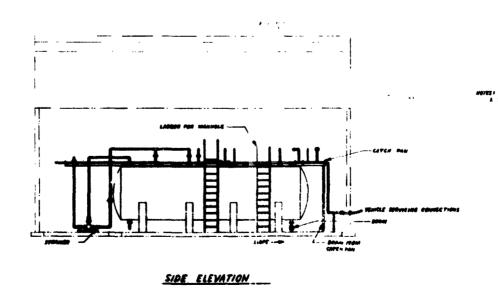






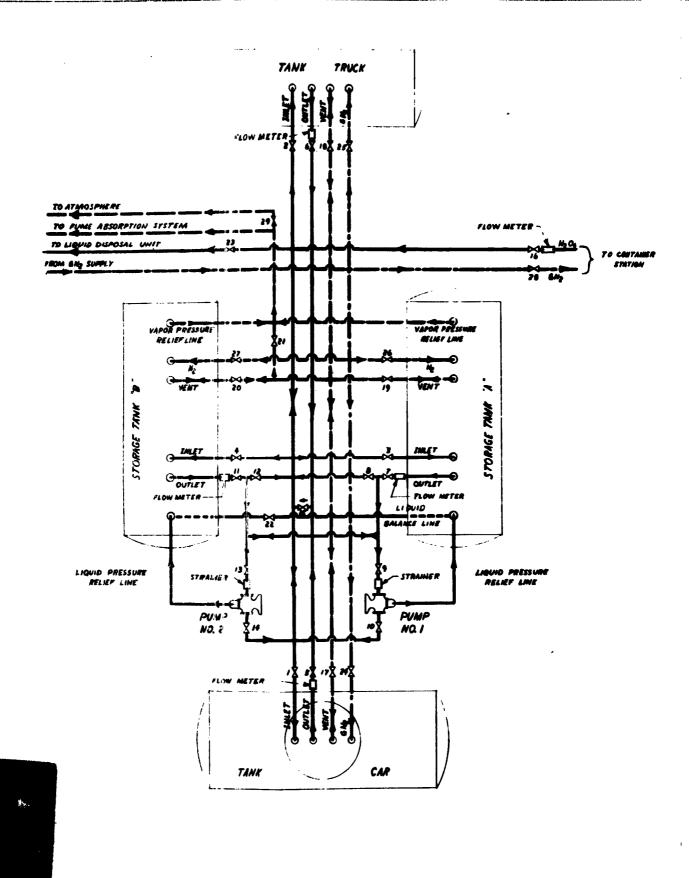
ISOMETRIC VIEW



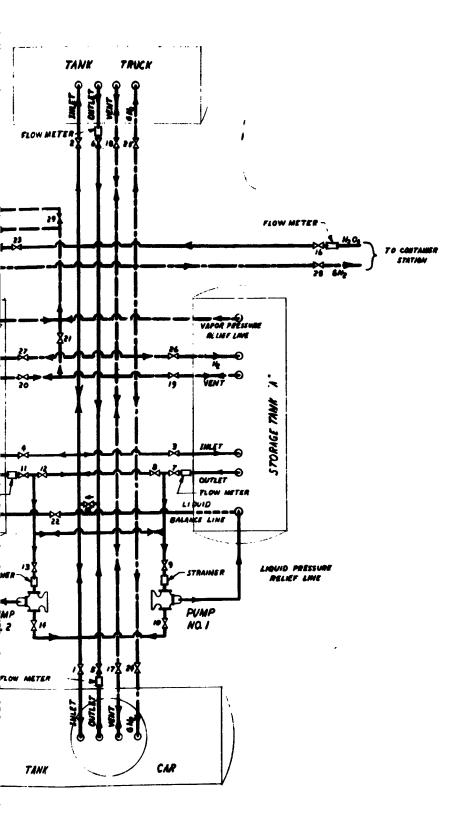


4.

E142-2-9 Storage and Transfer System



D142-2-10 Storage and Transfer System - Flow Diagram



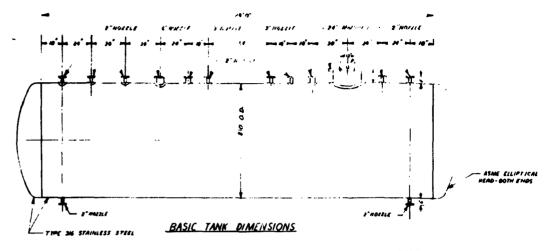
LEGEND

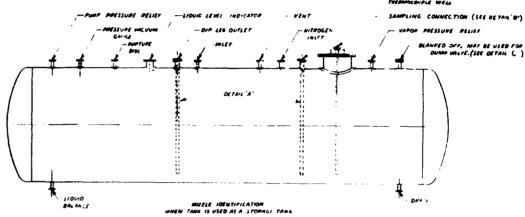
NITROGEN TETROXIDE LIQUID (Nº 04) LINES

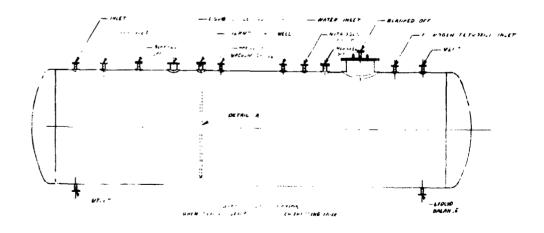
NITROGEN TETROXIDE VAFOR (Nº 04) LINES

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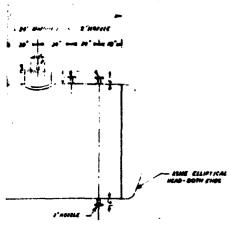


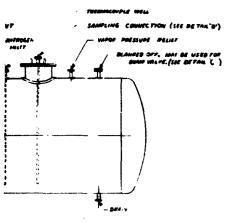


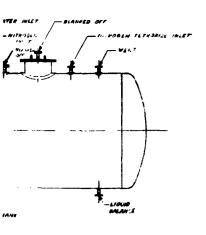


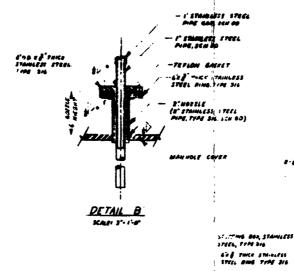


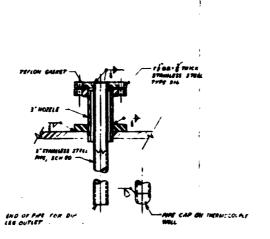




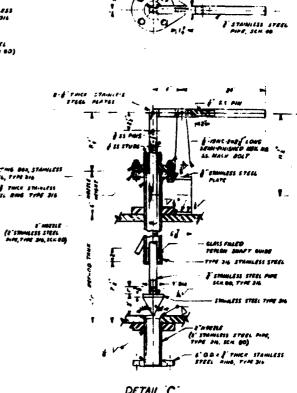












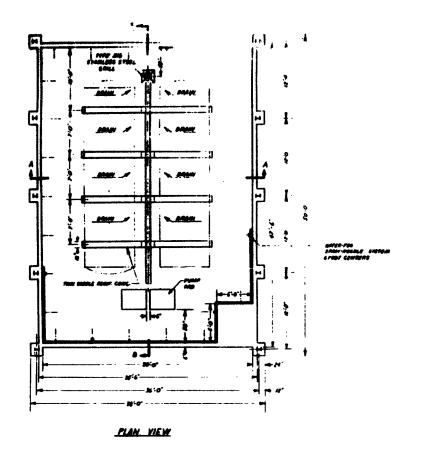
HALL STANGES. STEEL

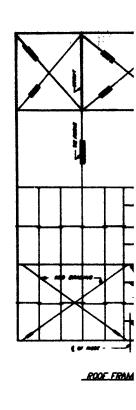
DETAIL C DAMP WALVE - OPTIONAL) SCALET 2"-1"-0"

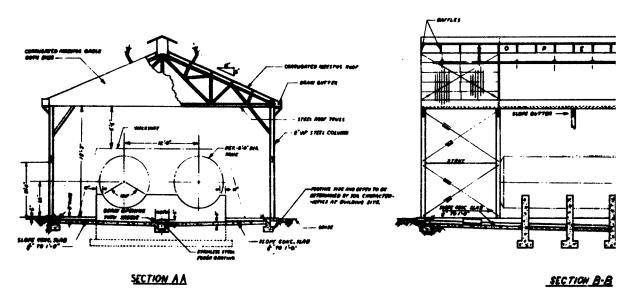
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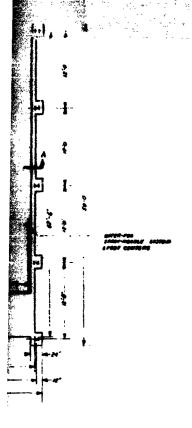


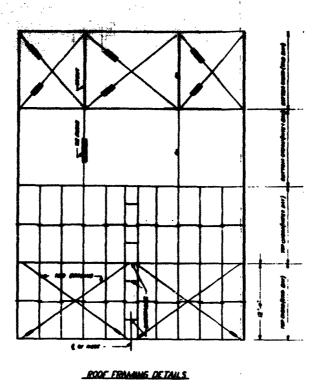


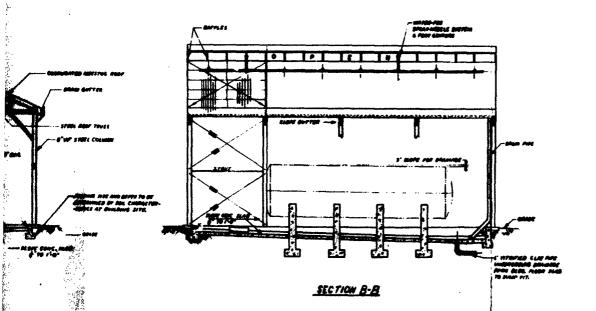




E142-2-12 Storage Tank Insta





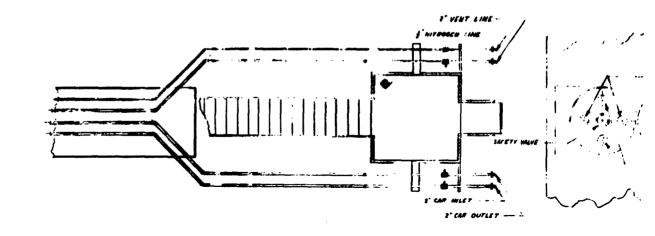


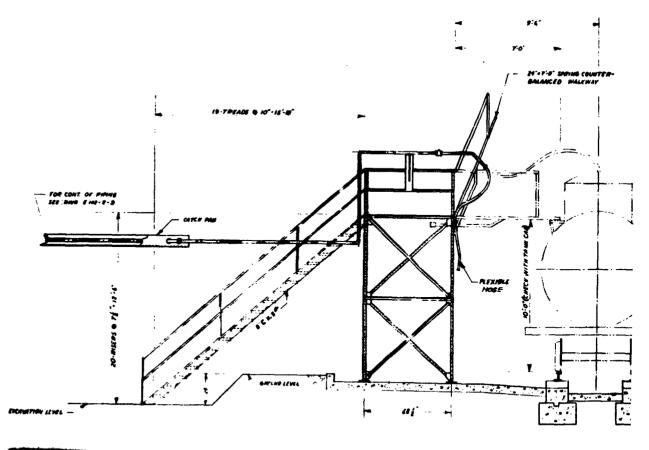
E142-2-12 Storage Tank Installation

GENERAL MOTES

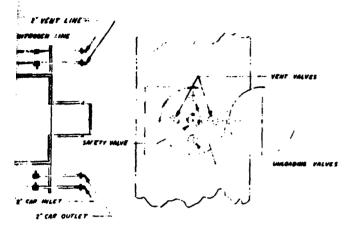
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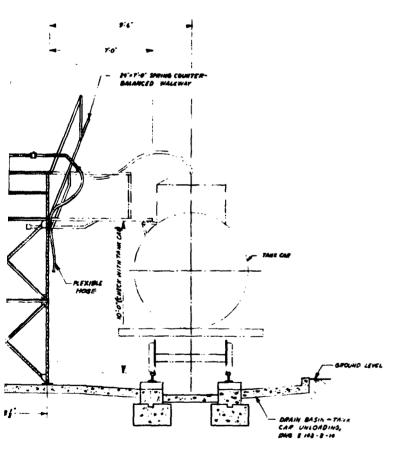














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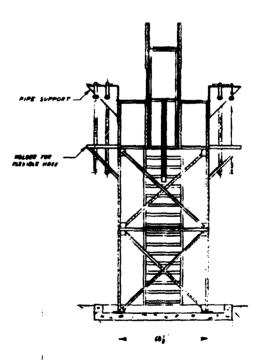
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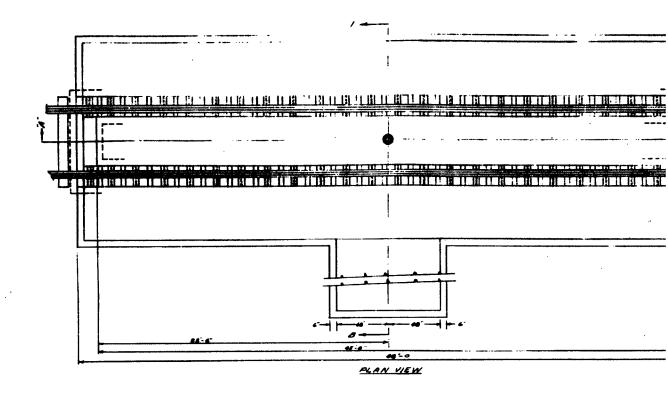
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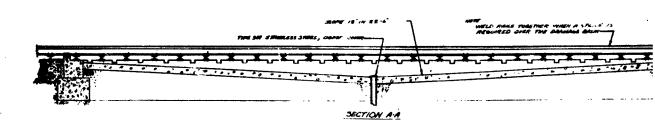
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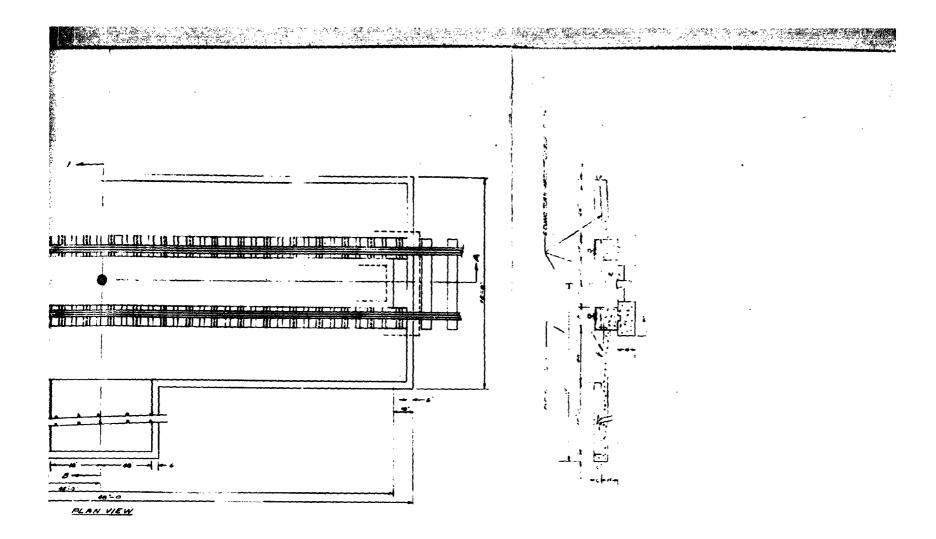


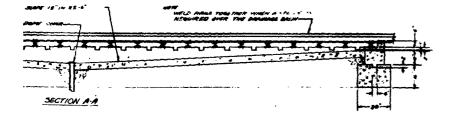




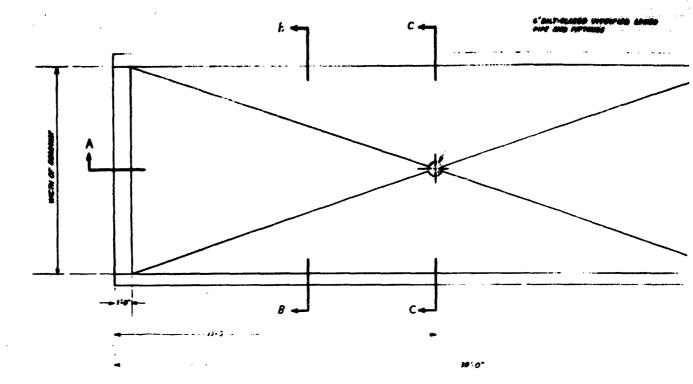




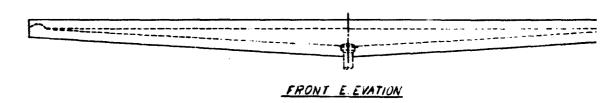


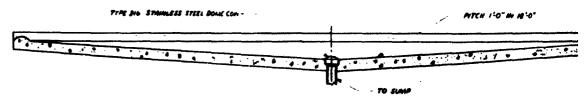




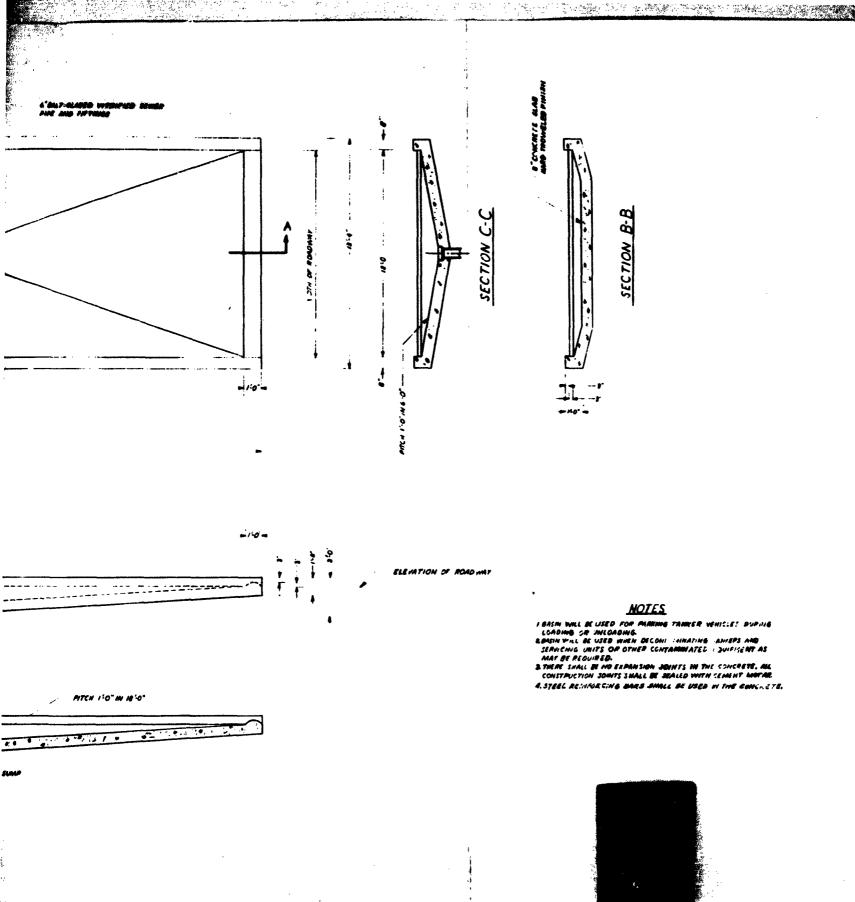


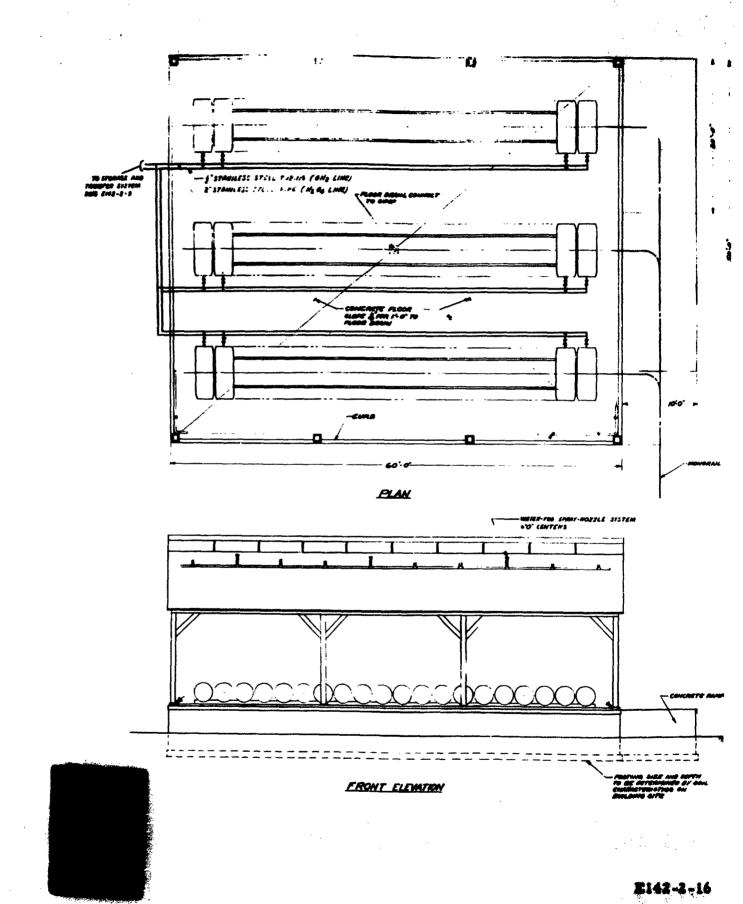
PLAN VIEW

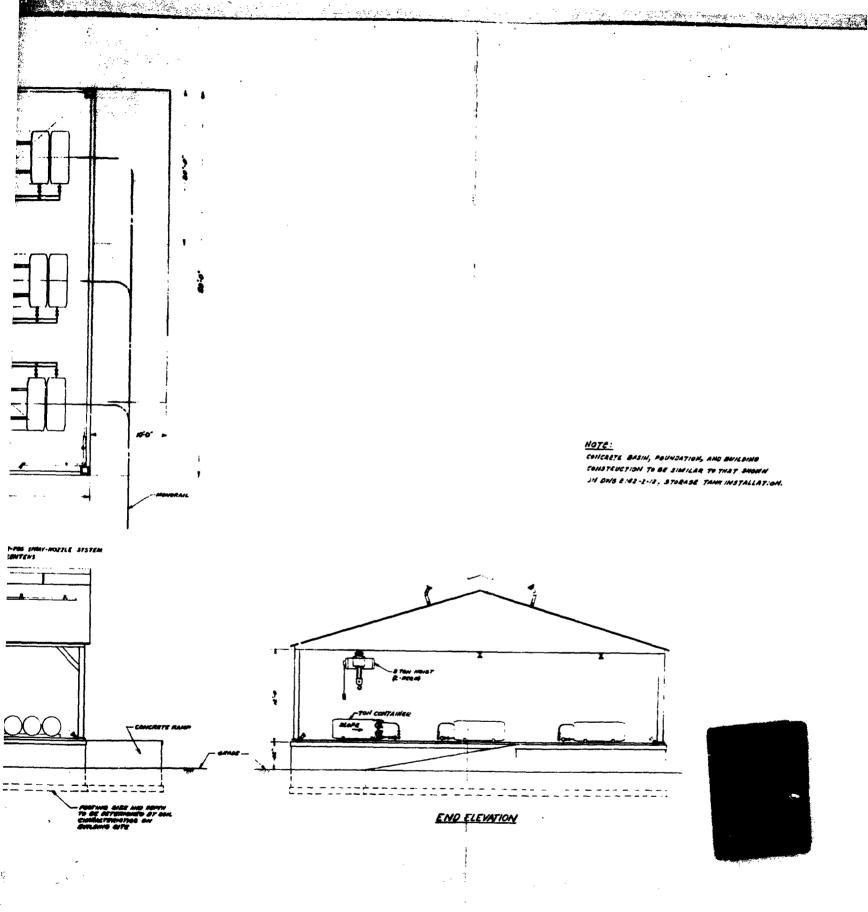




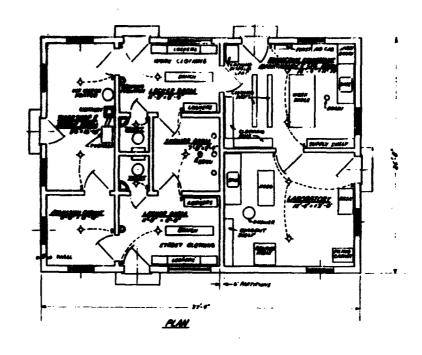
SECTION A-A

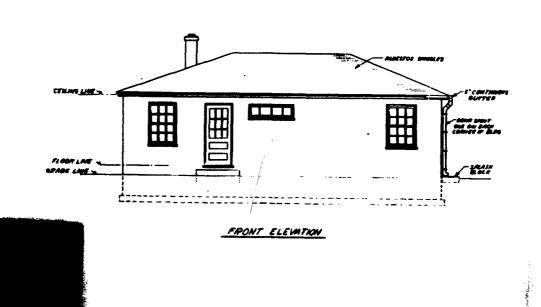


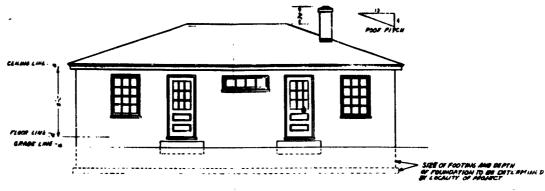




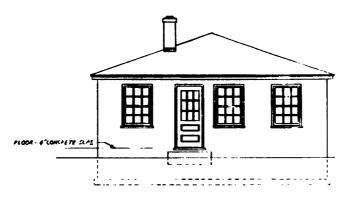
E142-2-16 Container Storage Installation



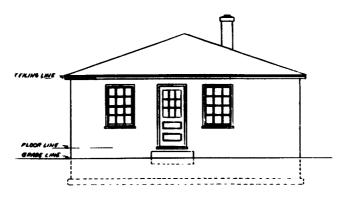




REAR ELEVATION

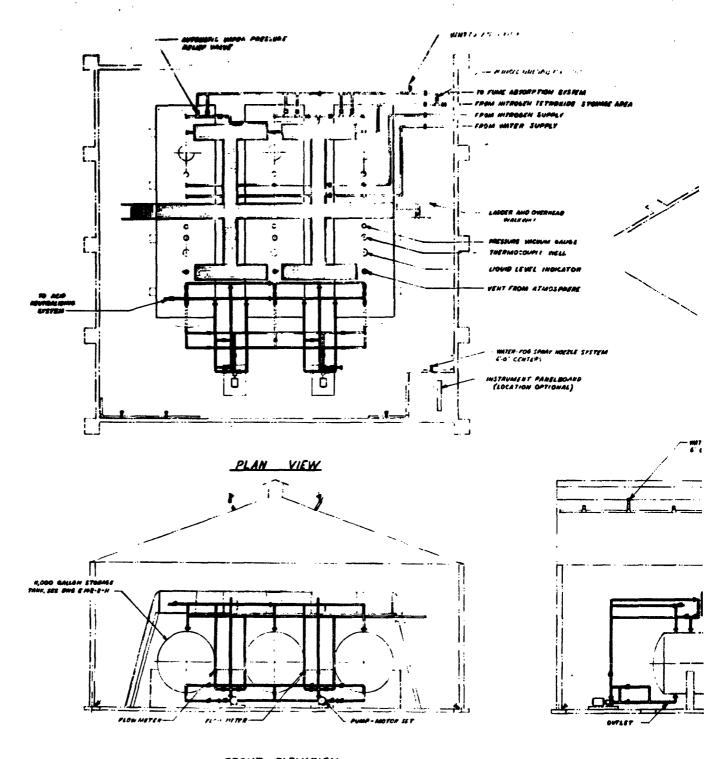


LEFT SIDE ELEVATION



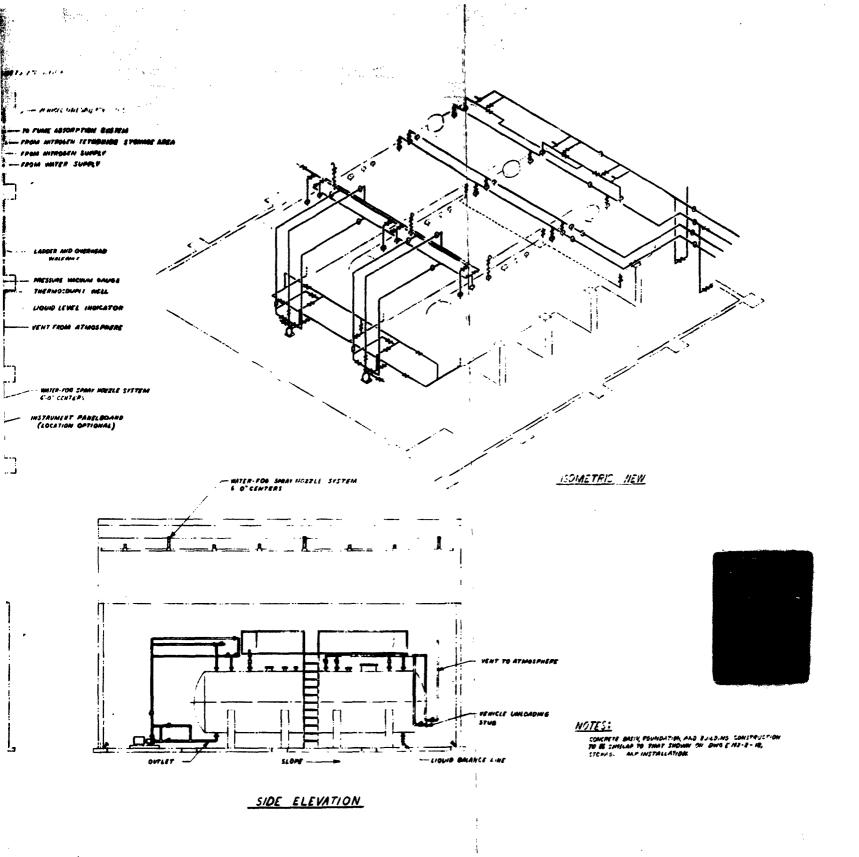
RIGHT SIDE ELEVATION

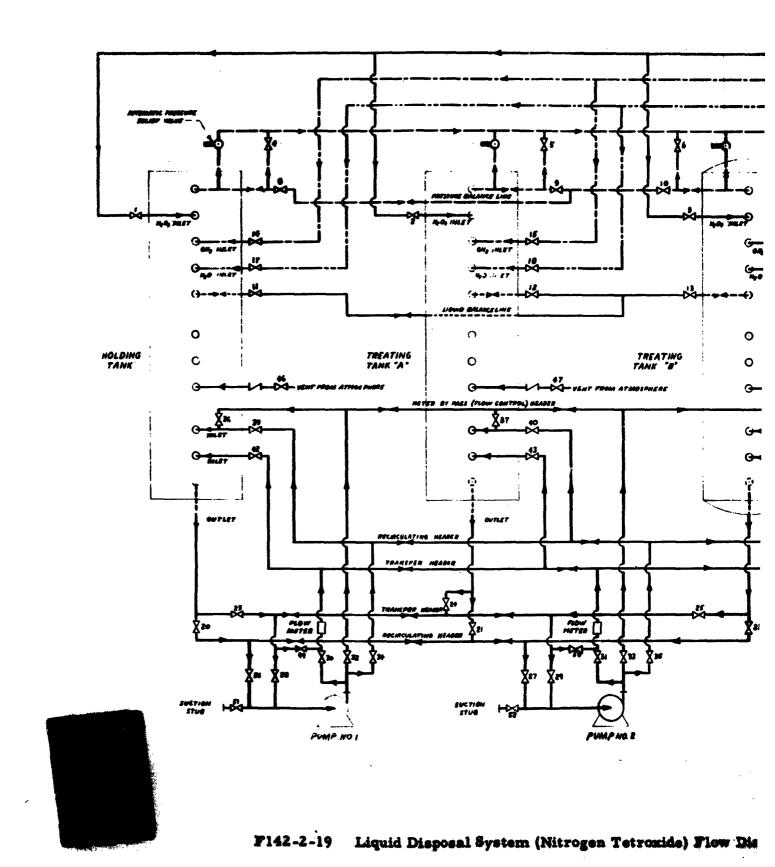


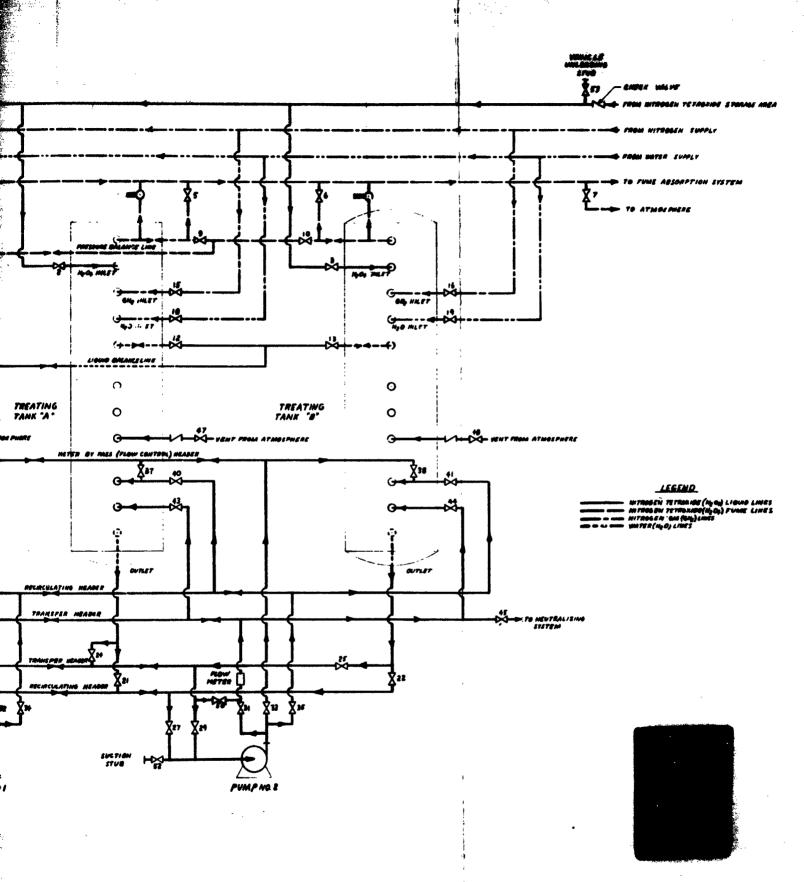


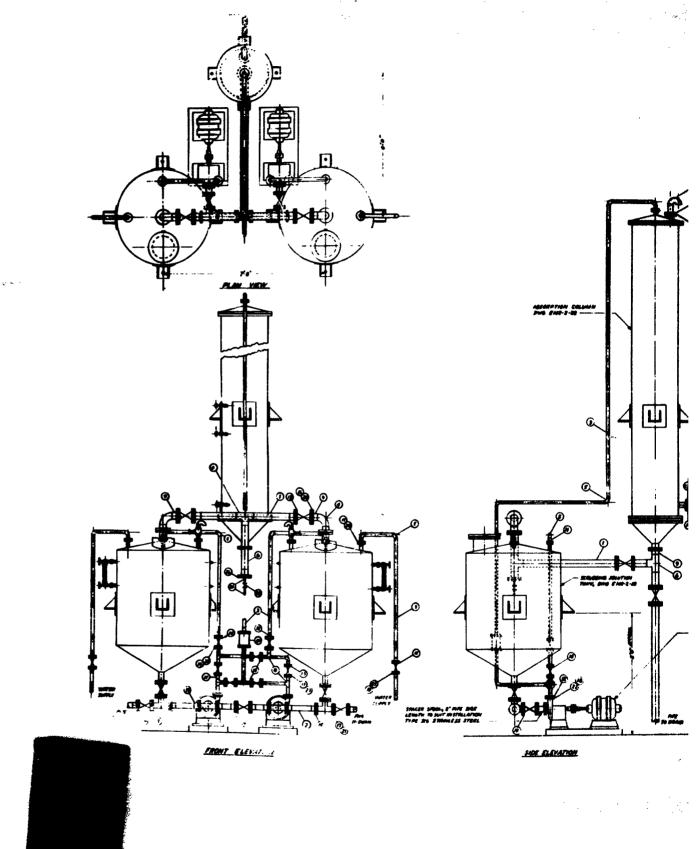
FRONT ELEVATION



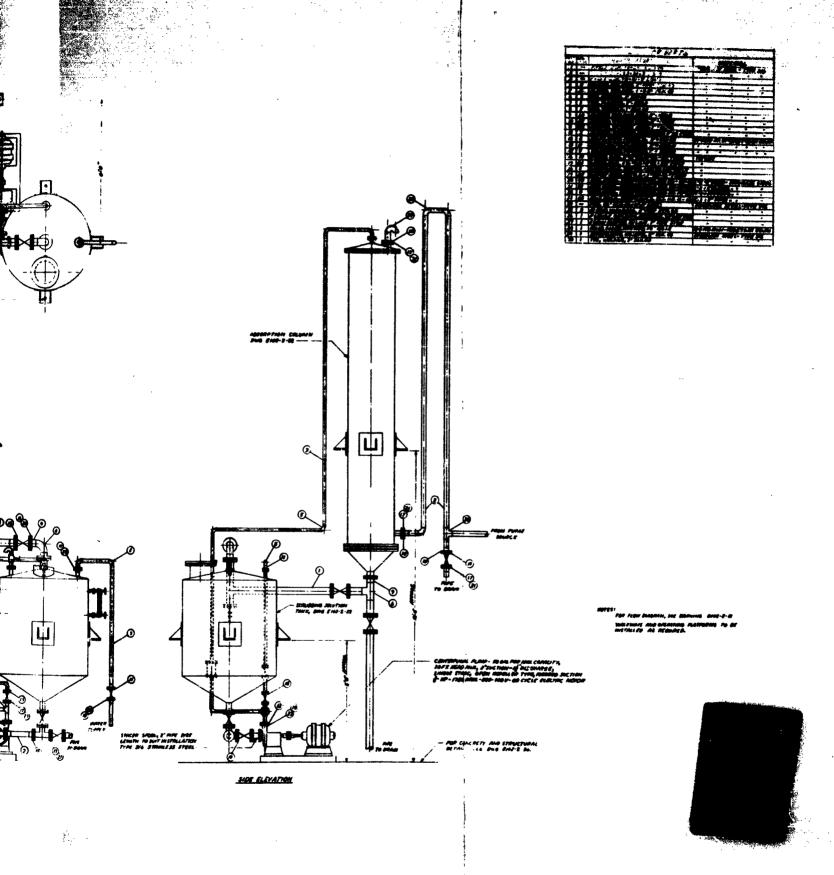


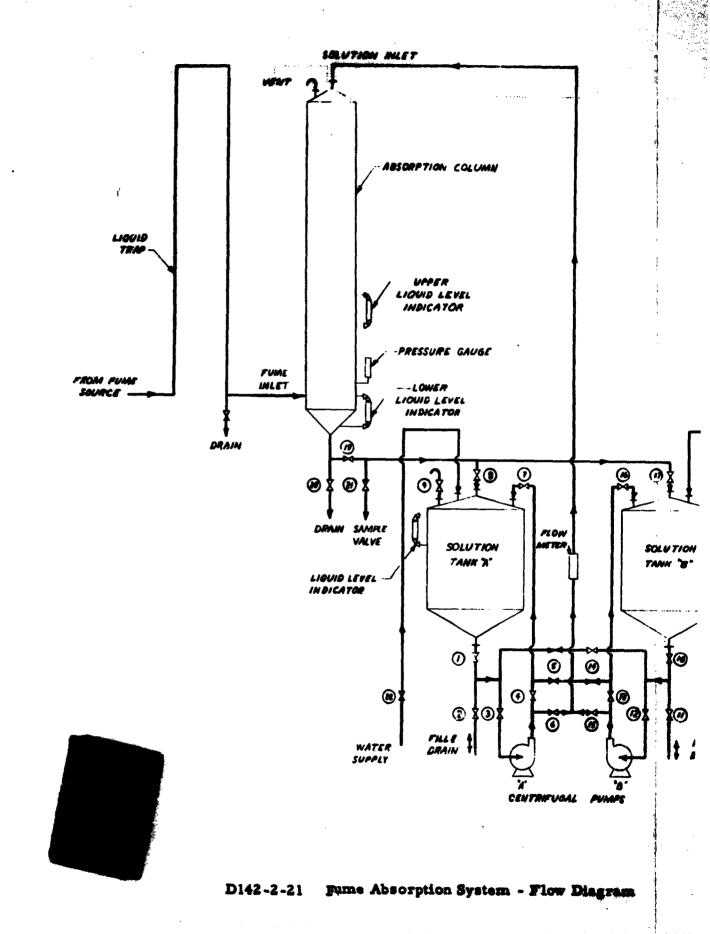


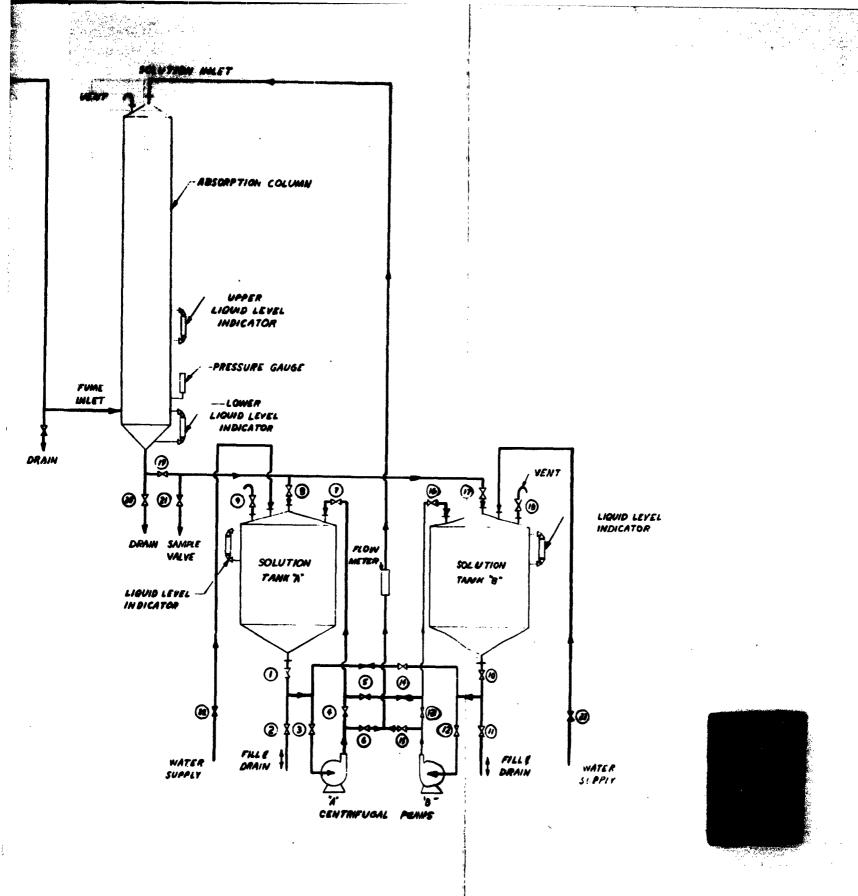




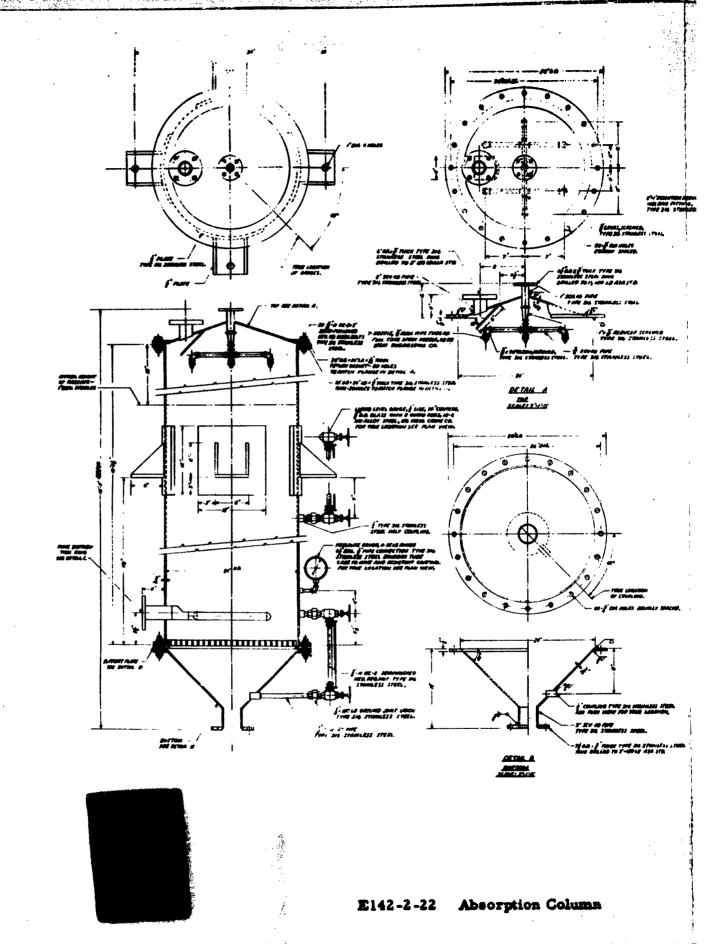
E142-2-20 Fume Absorption System

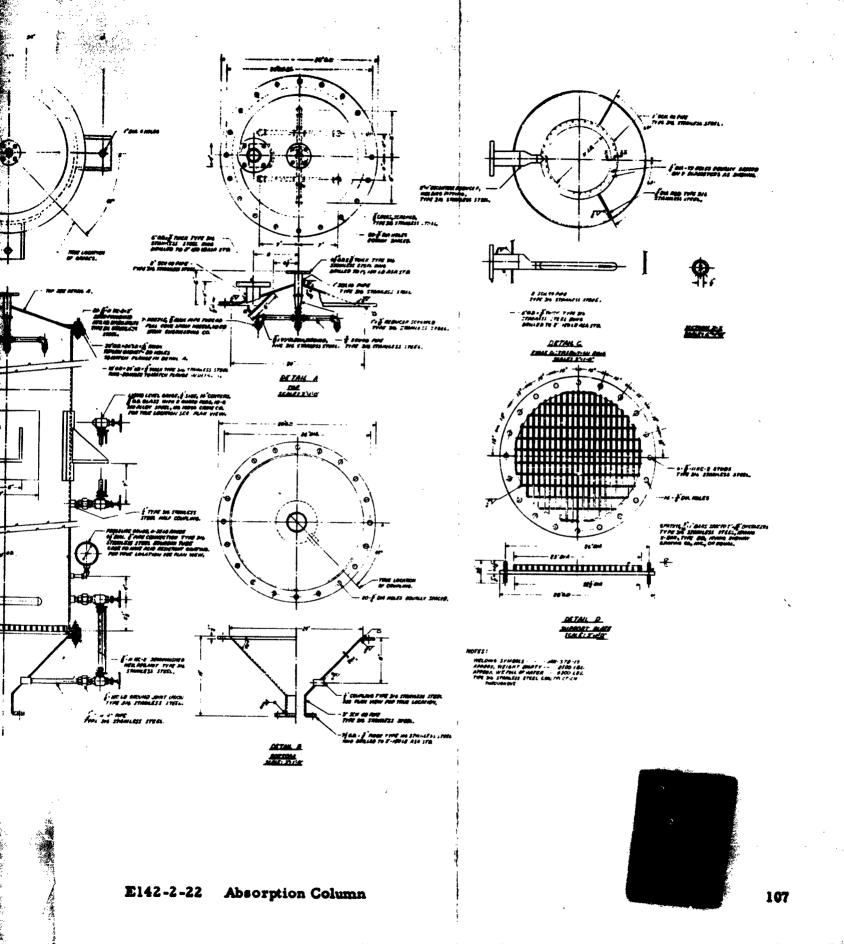


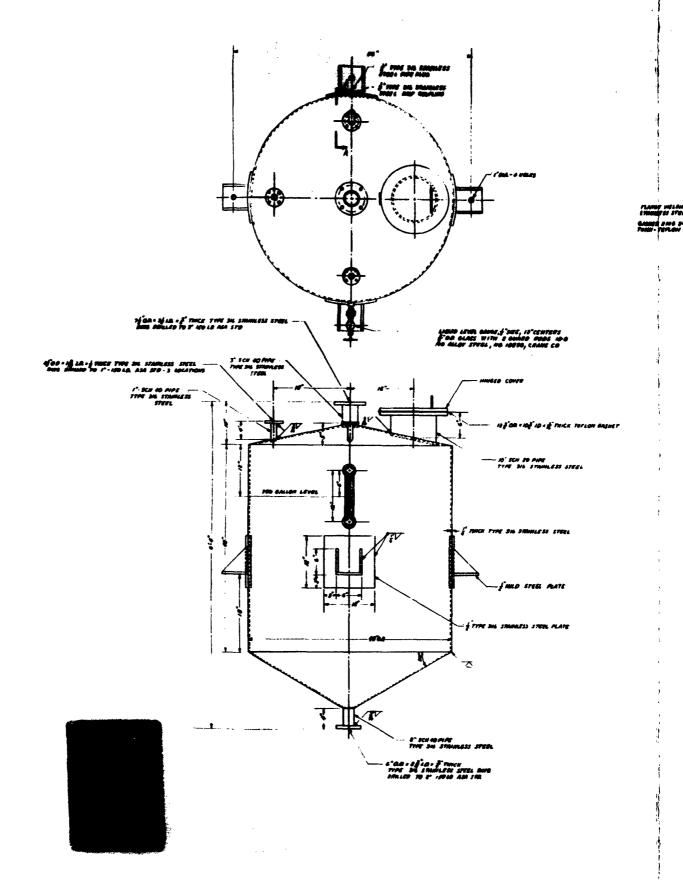




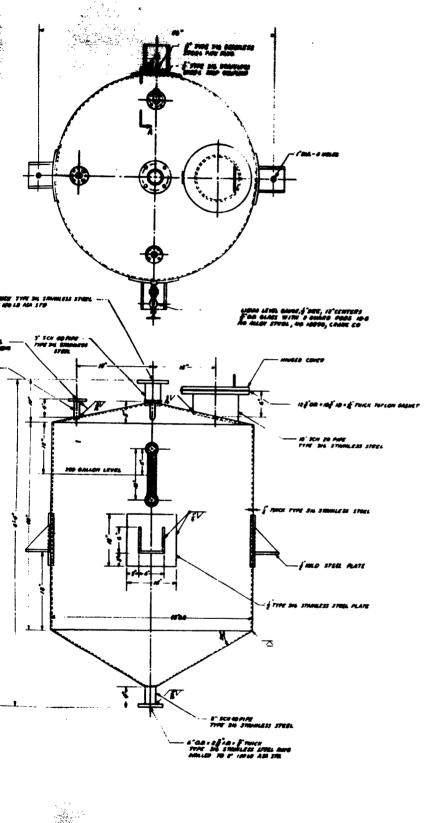
D142-2-21 Jume Absorption System - Flow Diagram

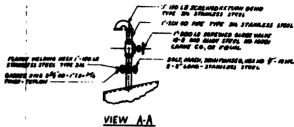




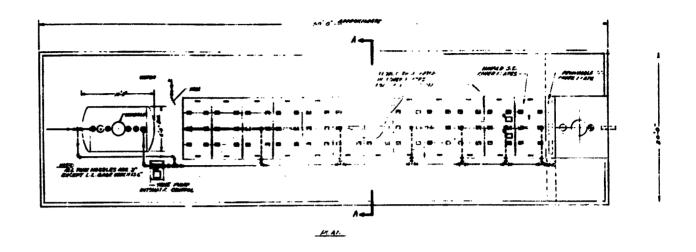


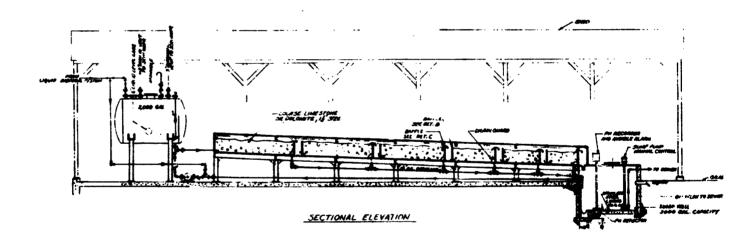
E142-2-23 Scrubbing Solution Tank (Potassium Permanganate)











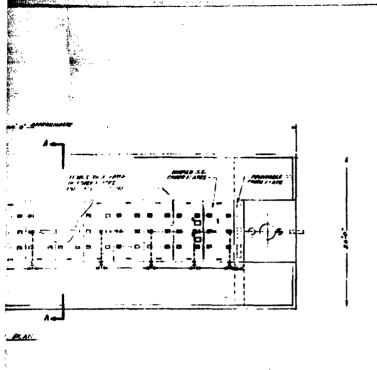
CETAL B - BAFFLE
STAINLESS STEEL
FRIGINES

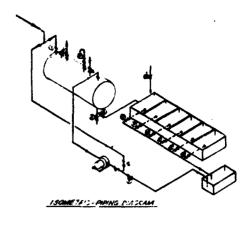
OETAN C. BAFFLE

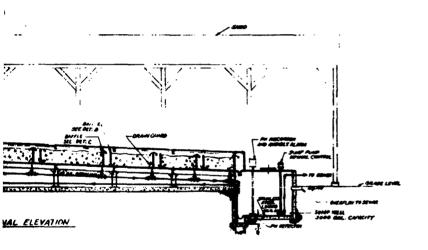
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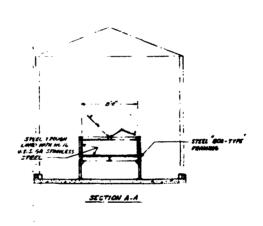
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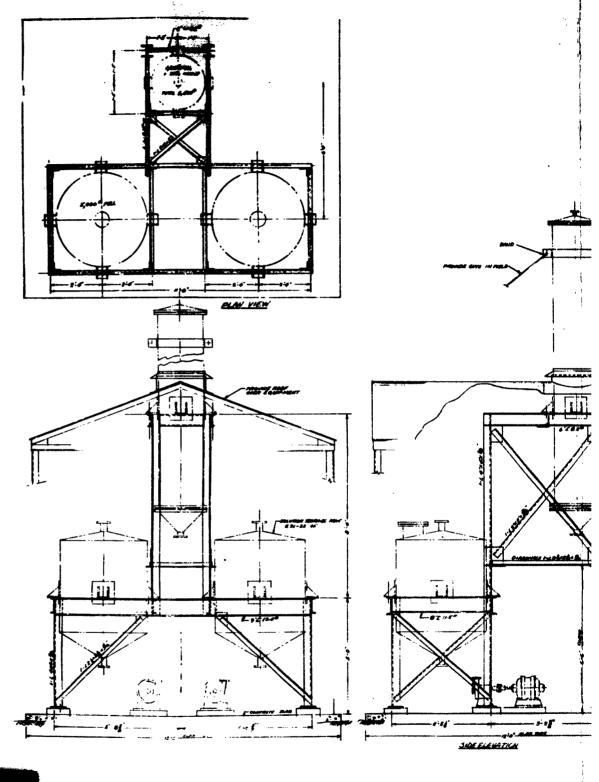






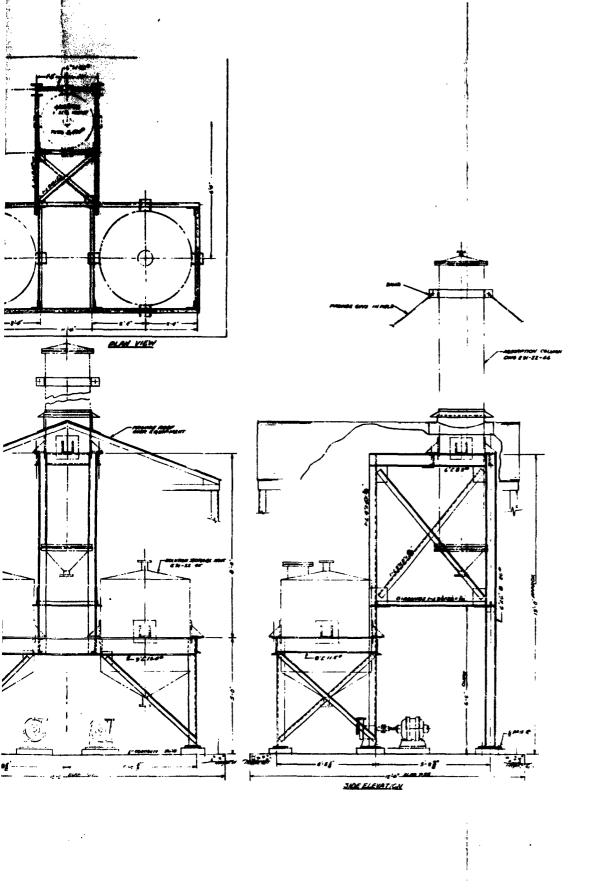
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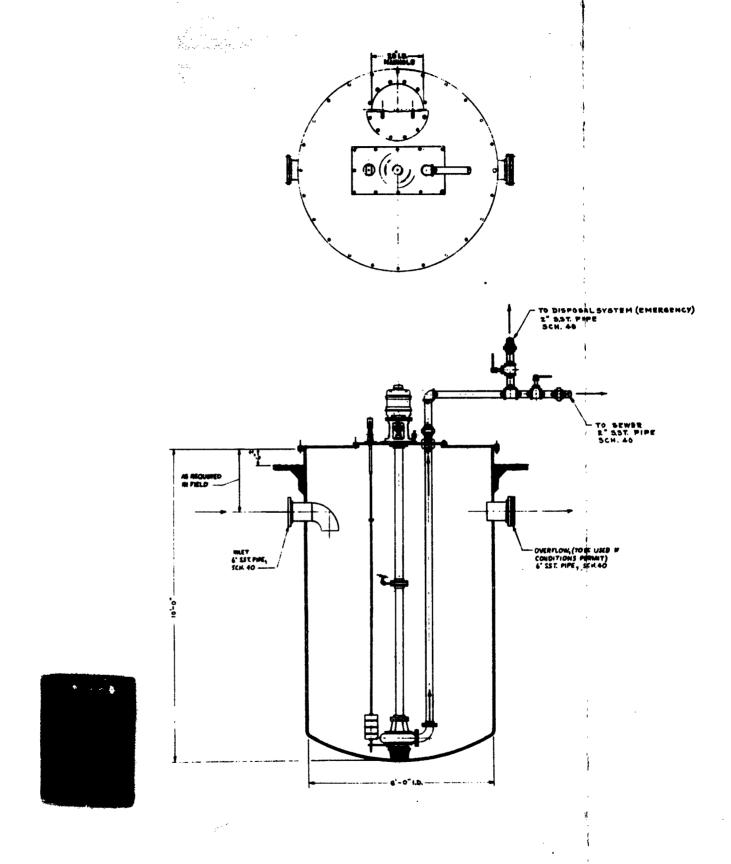
F142-2-25 Sump Pump Installation



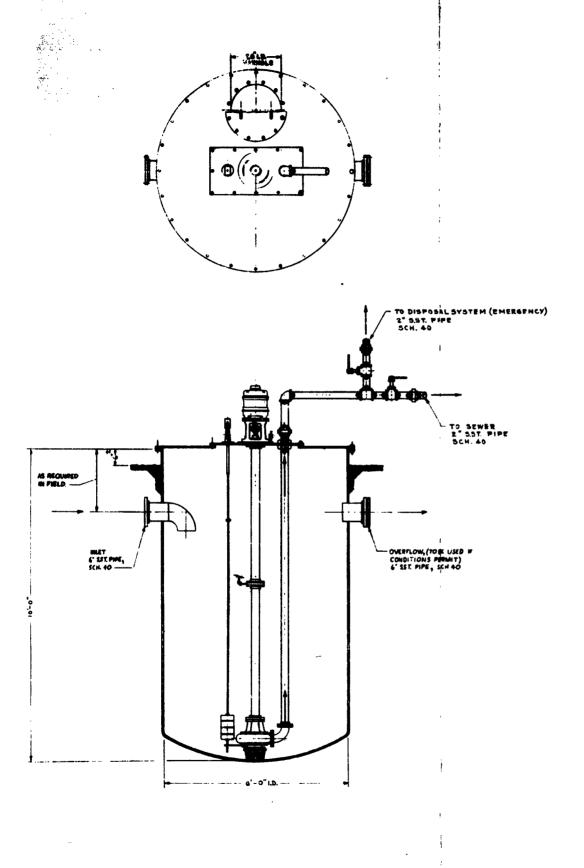
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F142-2-25 Sump Pump Installation



E142-2-26 Fume Absorption System - Structural Details





E142-2-26 Fume Absorption System - Structural Details

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